



HEADQUARTERS
U.S. ARMY ARMAMENT,
MUNITIONS AND CHEMICAL COMMAND

FINAL REPORT

DEVELOPMENT OF
METHODOLOGY AND TECHNOLOGY
FOR
IDENTIFYING AND QUANTIFYING
EMISSION PRODUCTS
FROM
OPEN BURNING AND OPEN DETONATION
THERMAL TREATMENT METHODS.

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BANGBOX TEST SERIES

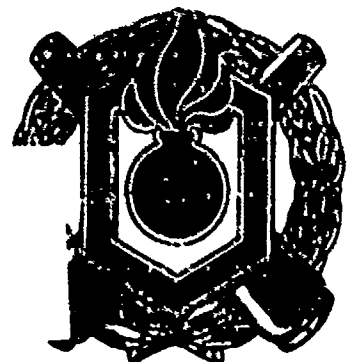
*VOLUME 2
TEST DEVELOPMENT*

JANUARY 1992

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19. ABSTRACT (Continue on reverse if necessary and identify by block number) The report covers in detail the test and the supporting data, and analyses used to answer the objectives. The supporting data and analyses are provided for the test development, selection of equipment and procedures, determination of BangBox volume, aerosol homogeneity, and the detonation products of TNT, the burn products of double-base propellant and composite propellant, and the detonation products of foam-attenuated TNT. All trials were accomplished in an air supported building (BangBox). Sampling was accomplished with real time instruments for CO ₂ , CO, NO _x , O ₂ , and SO ₂ ; canister samplers for SF ₆ , CO ₂ , CO, and volatile hydrocarbons; Quartz fiber filters with backup XAD-2 TM resin cartridges for semivolatile organics, and various instruments for size and mass of particulates. For each analyte measured an emission factor was calculated by the concentration times volume of the building and by the carbon balance method. The carbon balance method is based on the conservation of carbon in the detonation or burn and thus becomes the trace (Cont'd on reverse)					
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element used as the basis for determining emission factors. The mass balance of carbon in the TNT detonation was established in the BangBox with CO₂ contributing 97 percent of the total carbon, CO carbon (0.50 percent) semivolatile and non volatile organics carbon (0.57 percent), and soot (1.7 percent). The tracer method (carbon balance) permits an emission factor estimate from every parcel of sampled air regardless of the homogeneity of the cloud whereas the concentrations times cloud volume method is only good when the concentration is constant throughout the entire cloud volume and the cloud volume can be measured. The test was successful in providing a method for obtaining emission factors in open air testing, providing data to select samplers, and demonstrating the effectiveness of the supercritical fluid chromatography assay for open air detonations and burns of PEP material.

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FOREWORD

A Test Planning Directive to conduct the OB/OD test in support of U.S. Army Armament, Munitions and Chemical Command (AMCCOM) was issued by U.S. Army Test and Evaluation Command (TECOM) on 28 April 1988¹. This test was conducted following the Technical Steering Committee Symposium which was convened in July 1988. The requirement for identifying and quantifying emissions from the open detonation of explosives and open burning of propellants was discussed in detail by authorities from throughout the military, academic, and commercial communities. Conclusions and recommendations developed during the symposium are reported in proceedings of the symposium².

The BangBox Test series report includes three volumes:

Volume 1. A summary which describes the planning phase, the conduct of trials, sample analyses and results, and the conclusions and recommendations. It is useful for those who need the background, synopsized results, conclusions, and recommendations without the complete details with the supporting data and information.

Volume 2. A stand-alone document which covers the detail of the complete test. It describes the test development, description of the test materiel, and the trial results as they relate to the test objectives and the explosives and propellants tested.

Volume 3. The quality assurance/quality control (QA/QC) report covers the QA/QC plan, detailed test plan, the letters of instruction (prepared for procedural instruction), the quality audits, the reports of the quality audits, and the results of the blind spikes analyzed by the laboratories.

¹Letter, AMSTE-TA-F, Headquarters, U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland, 20 April 1988, subject: Test Planning Directive for Special Study of Open Burning/Open Detonation (OB/OD), Phase II, TECOM Project No 2-CO-210-000-017.

²*Proceedings of the Technical Steering Committee Symposium 6-8 July 1988*, Headquarters, United States Army Armament, Munitions and Chemical Command, Rock Island, Illinois, August 1991.

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Mr. Robert A. Cary of Sunset Laboratory, Forest Grove, Oregon, provided thermal analysis of filter samples for organic, elemental, and inorganic carbon.

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SECTION 1. BANGBOX TEST DEVELOPMENT

1.1. Objectives

The purpose of the open burning/open detonation (OB/OD) BangBox (BB) Test was to verify and validate OB/OD test technology including:

- 1.1.1. Instruments, sampling equipment, and procedures being considered for subsequent use aboard a fixed-wing aircraft (FWAC) on larger-scale outdoor field trials;
- 1.1.2. Procedures for transport and storage of sample specimens;
- 1.1.3. Sample assay techniques;
- 1.1.4. Quality assurance/quality control (QA/QC) procedures; and
- 1.1.5. The proposed carbon balance method of determining concentrations/emission factors of OB/OD combustion products.

1.2. Testing Authority

A Test Planning Directive to conduct the OB/OD test in support of U.S. Army Armament, Munitions and Chemical Command (AMCCOM) was issued by U.S. Army Test and Evaluation Command (TECOM) on 28 April 1988.

1.3. Test Concept

1.3.1. This test, conducted at Sandia National Laboratories (SNL) from 28 November 1988 through 16 February 1989, included 12 days of testing activity. The first goal was to characterize the BB test facility so that data could be analyzed in relation to the environment in which it was obtained. Following characterization trials, the test progressed to recording raw real-time and near-real-time data for gaseous pollutants, and trapping trace organic (exotic) compounds produced by the small-

scale OB of propellants and OD of explosives. Target analytes were originally selected at a July 1988 symposium by members of the technical steering committee (TSC) with expertise in chemistry, sampling, spectrometry-chromatography, explosives, propellants, and environmental regulations. The group proposed a list of 71 target analytes for consideration in developing analysis plans for OB/OD trial samples. As analysis results from the initial BB trinitrotoluene (TNT) detonation and propellant burn samples became available, this original list was refined, with some analytes being withdrawn and others added. The revised analyte list for TNT detonations, double-base propellant burns, and for composite propellant burns is presented in Table 1.1.a, b, and c. Analysis of the combination of characterization data, criteria pollutant data, and results of chemical assay provided the baseline data that was the main goal of this test.

1.3.2. The following were determined from the data from this series of trials:

1.3.2.1. The BB's volume;

1.3.2.2. The BB's ventilation (leak) rate (and consequential atmospheric dilution) when the inflation blower was operating;

1.3.2.3. The capability of achieving gaseous homogeneity within the BB by using large mixing fans;

1.3.2.4. The ability of candidate equipment and procedures to accurately capture and characterize representative samples of detonation emissions (particularly trace organic combustion products) following detonation of small explosive charges;

1.3.2.5. The ability of candidate equipment and procedures to accurately capture and characterize representative samples of burn emissions (particularly trace organic combustion products) following ignition of small quantities of assorted propellants; and

1.3.2.6. The suitability of foam to serve as a soil surrogate during BB trials involving detonation of explosives.

Table 1.1a Target Analytes for the OB/OD BangBox Test Series.

Analyte	Trial		
	TNT Detonation	Double-base Propellant Burn	Composite Propellant Burn
Carbon monoxide	X	X	X
Carbon dioxide	X	X	X
Ozone	X	X	X
Nitrogen oxides (NO, NO ₂ , NO _x)	X	X	X
Sulphur dioxide	X	X	X
Hydrogen cyanide			X
Hydrogen chloride			X
Total suspended particulate	X	X	X
PM ₁₀ particulate	X	X	X
C ₁ to C ₁₀ hydrocarbons ^a	X	X	X
2,4-Dinitrotoluene	X	X	X
2,6-Dinitrotoluene	X	X	X
4-Nitrophenol	X	X	X
Phenol	X	X	X
Toluene		X	
Benzene	X	X	X
2,4,6-Trinitrotoluene	X	X	X
1,3,5-Trinitrobenzene	X		
Resorcinol			X
Salicylic acid		X	
Nitroglycerine		X	
Triacetin		X	
Di-n-propyl adipate		X	
Phenyl disodecyl phosphite			X
Dioctyl sebacate			X
2,2 Methylene bis-(4-methyl)-6-t-butylphenol			X
5-Ethyl-1,3-diglycidyl-5-methyl hydantoin diepoxide			X
Diethylenetriamine			X
Benz[a]anthracene	X	X	X
Benzo[a]pyrene	X	X	X

Table 1.1b Target Analyte List for the OB/OD BangBox Test Series - Cont'd.

Analyte	Trial		
	TNT Detonation	Double-base Propellant Burn	Composite Propellant Burn
2-Naphthylamine	X	X	X
Dibenz[a,h]anthracene	X	X	X
2-Methylnaphthalene	X		
Naphthalene	X	X	X
Pyrene	X		
1-Methylnaphthalene	X		
Biphenyl	X		
Phenanthrene	X		
Benz[c]acridine	X	X	X
Dibenzofuran		X	X
1,6-Dinitropyrene	X	X	X
2-Nitronaphthalene	X	X	X
1-Nitropyrene	X	X	X
4-Nitrosodiphenylamine		X	
2-Nitrodiphenylamine		X	
4-Nitrodiphenylamine		X	
N-Nitrosodiphenylamine	X	X	X
Polychlorinated dibenzo-dioxins			X
Polychlorinated dibenzo-furans			X
Diphenylamine		X	
Isophorone diisocyanate			X
2,5-Diphenyloxazole	X		
1,1,3-Trimethyl-3-phenylindane	X		

Table 1.1c Target Analyte List for the OB/OD BangBox Test Series - Cont'd.

Analyte	Trial		
	TNT Detonation	Double-base Propellant Burn	Composite Propellant Burn
Antimony	X	X	X
Arsenic	X	X	X
Barium	X	X	X
Cadmium	X	X	X
Chromium	X	X	X
Lead	X	X	X
Beryllium	X	X	X
Mercury	X	X	X
Nickel	X	X	X
Fluoride ion	X	X	X
Nitrate ion	X	X	X

*For the complete list of C₁ to C₁₀ hydrocarbons (grouped as paraffins, olefins, non-benzene aromatics, and terpenes) analyzed by Oregon Graduate Center (OGC) from 6-L canister samples, see Table 5.7.

1.4. Description of Test Materiel

1.4.1. Explosives

1.4.1.1. Explosives used during this test were 227gram (0.5-lb) blocks of TNT, from which all wrapping material had been removed. This combination of type (TNT) and quantity of explosive was selected to provide a "worst-case" scenario, i.e., an explosive known to produce relatively large quantities of pollutants for a given source mass due to TNT's 74 percent negative oxygen balance (quantity of oxygen needed per 100 g of TNT to completely convert C, H, N to stable oxides), and a quantity small enough to ensure a detonation of less-than-maximum efficiency. This generated a spectrum of pollutants and emissions representative of those produced during large-scale OD operations and provided a meaningful challenge to real-time analyzers, collection devices, and chemical laboratory analysis procedures.

1.4.1.2. Each explosive charge consisted of pressed TNT grains ranging in size from -35 to +65 mesh. Graphite, used to facilitate the pressing process, was the only other component in the blocks. Initiators (one per block of TNT) were RP83 detonators consisting of pentaerythritol tetranitrate (PETN) and hexamethylenetrinitramine (RDX) enclosed in an aluminum alloy casing. The detonators were electrically connected to a remote firing mechanism by two #16 insulated and tinned copper wires. TNT was provided by the U.S. Army, and initiators were provided by SNL.

1.4.2. Propellants

1.4.2.1. Both double-base and composite propellants were burned during this test. The double-base propellant consisted primarily of nitrocellulose (51 percent) and nitroglycerine (approximately 39 percent). The composite propellant was composed primarily of ammonium perchlorate (AP) (approximately 85 percent) and hydroxyl-terminated polybutadiene (8 percent). Both propellants had small amounts of additional chemicals used for purposes such as stability, burning rate modification, physical property enhancement, or as a processing aid. The propellant samples were ignited with an electric match. Propellants were provided by the U.S. Navy, and igniters were provided by SNL.

1.4.3. Test Facility

All testing was conducted at SNL, Kirtland Air Force Base, Albuquerque, New Mexico, in an enclosed, air-supported hemispherical building with a rigid air lock. This building is locally referred to as the BB.

1.4.4. Detection/Sampling Instrumentation and Collection Devices

1.4.4.1. Particulate Detectors and Samplers

1.4.4.1.1 TSI Differential Mobility Particle Sizer (DMPS) System: 0.01- to 0.5- μm diameter.

1.4.4.1.2 TSI Aerodynamic Particle Sizer (APS) System: 0.5- to 15- μm diameter.

1.4.4.1.3 Teflon™ filter: 47-mm diameter, 2- μ m pore size; Gelman, R2PJ047.

1.4.4.1.4 Nuclepore™ Filter: 0.2- μ m pore size, 47-mm diameter, Nuclepore™ Part No. 181106, to collect particulate samples for characterization by scanning electron microscopy (SEM).

1.4.4.1.5 Aerosol probe: 0.15- to 3.0- μ m diameter, Particle Measuring Systems, Inc. (PMS) Active Scattering Aerosol Spectrometer Probe (ASASP)-100X.

1.4.4.1.6 Aerosol Probe: 2- to 47- μ m diameter, PMS Forward Scattering Spectrometer Probe (FSSP)-100X.

1.4.4.1.7 Integrating Nephelometer: Belfort Industries Model 1590.

1.4.4.2. Total Hydrocarbon (THC) Detectors

1.4.4.2.1 Flame ionization detector (FID): 0.5 ppm detection limit, Century Organic Vapor Analyzer 128 (characterization trials only).

1.4.4.2.2 Photoionization detector (PID): ultraviolet (UV) lamp, 0.1 ppm detection limit, HNU Instruments Model PI-101 (characterization trials only).

1.4.4.3. Gas Analyzers

1.4.4.3.1 Gas Filter Correlation CO₂ Analyzer, Thermo Electron Instruments (TECO) Model 41H.

1.4.4.3.2 Gas Filter Correlation CO Analyzer, TECO Model 48.

1.4.4.3.3 Pulsed Fluorescence SO₂ Analyzer, TECO Model 43.

1.4.4.3.4 UV Photometric O₃ Analyzer, TECO Model 49.

1.4.4.3.5 Chemiluminescent Nitrogen Oxides (NO_x) Analyzer, Columbia Scientific Instruments

(CSI) Model 1600.

1.4.4.3.6 Sulfur hexafluoride (SF_6) analyzer, Shimadzu gas chromatograph (GC) with electron capture detector (ECD).

1.4.4.3.7 Bubblers for HCN, HCl, and NH_3 .

1.4.4.4. Semivolatile organic sampling train (semi-VOST) Trace Organic Material Samplers

1.4.4.4.1 Modified resin cartridge train (Andersen PS-1), with two resin cartridges.

1.4.4.4.2 Quartz-fiber filters.

1.4.4.5. Samplers for Metals: TeflonTM filters, 47-mm diameter, 2- μm pore size; Membrana, Inc.

1.4.4.6. Collectors

1.4.4.6.1 VelostatTM bag, 1.5- m^3 .

1.4.4.6.2 Passivated stainless steel canisters (SSC), 6-L.

1.4.4.6.3 Stainless steel (SS) tanks, 32-L.

1.4.4.7. Ancillary Equipment

1.4.4.7.1 Two mixing fans, 250- m^3/min (8,600 ft^3/min) floor-stand fans, Granger Electric.

1.4.4.7.2 Color video camera.

1.4.4.7.3 Camera, video, high-speed (HS), 200 frames/s, NAC Corporation.

1.4.4.7.4 Camera, HS, 500 to 10,000 frames/s (5000 frames/s was speed used).

1.4.4.7.5 Pumps. for semi-VOST. GAST Industries, Carbon Vane.

1.4.4.7.6 Electronic mass flowmeters, for resin trains and sample filters, Matheson Model SEF-1454.

1.4.4.8. Detector, Sampler, Collector and Ancillary Equipment Citing: The location of detectors, samplers, collectors, and ancillary equipment are listed below.

1.4.4.8.1 BB airlock: Instruments are listed in Paragraphs 1.4.4.1.1 through 1.4.4.1.4, 1.4.4.2, 1.4.4.3.1 through 1.4.4.3.5, 1.4.4.4, 1.4.4.5, 1.4.4.6, and 1.4.4.7.5 and 1.4.4.7.6. The physical arrangement and sampling connections of the instruments and equipment, at the time the first trial was conducted, are schematically represented in Figure 1.1.

1.4.4.8.2 BB chamber: Instruments are listed in Paragraphs 1.4.4.1.5 through 1.4.4.1.7, 1.4.4.3.7, 1.4.4.4, 1.4.4.5, 1.4.4.6.3, and 1.4.4.7. The physical arrangement of the instruments and equipment, at the time the first trial was conducted, is schematically represented in Figure 1.2.

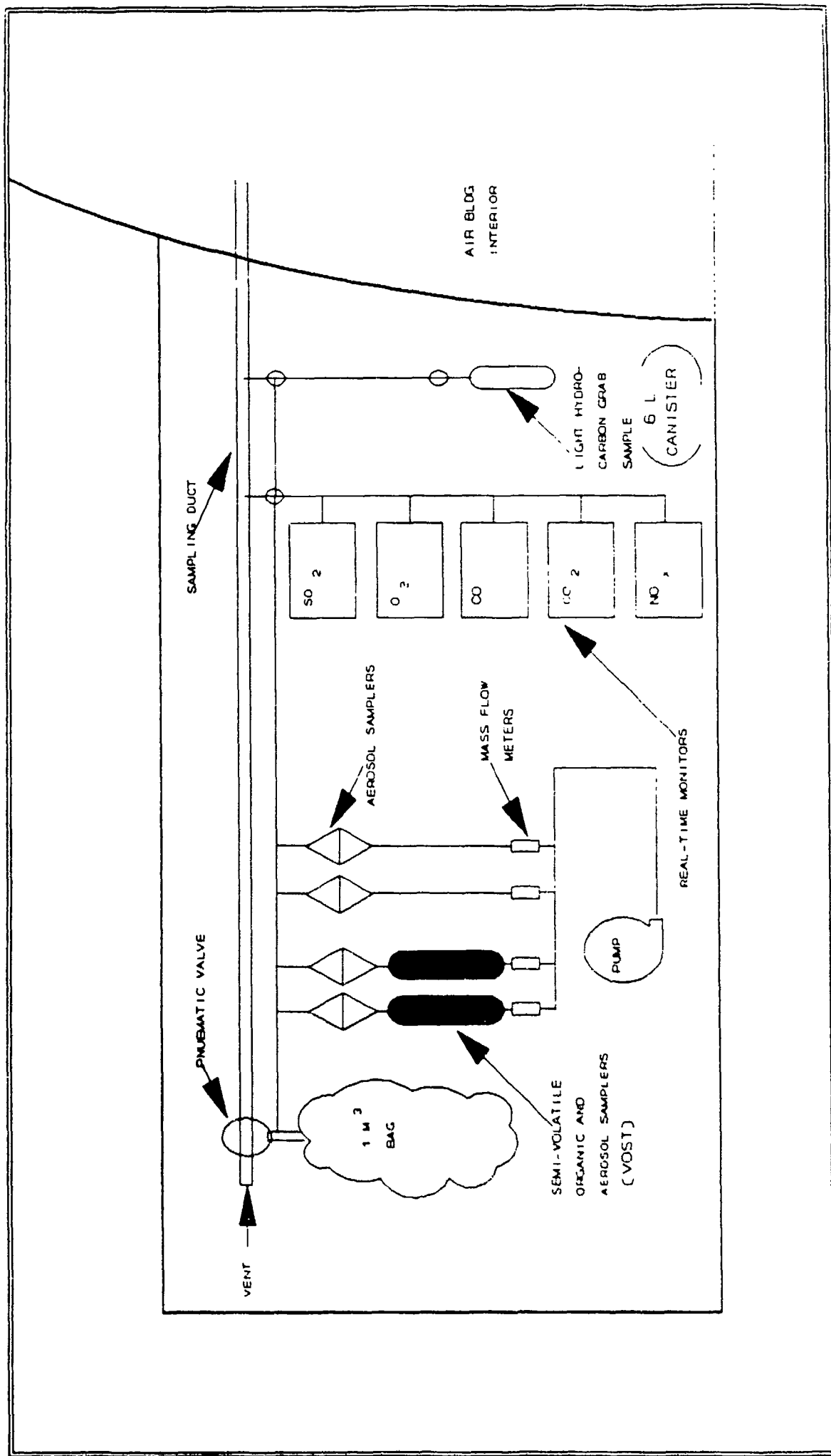


Figure 1.1 BangBox Airlock Instrumentation and Sampling Equipment.

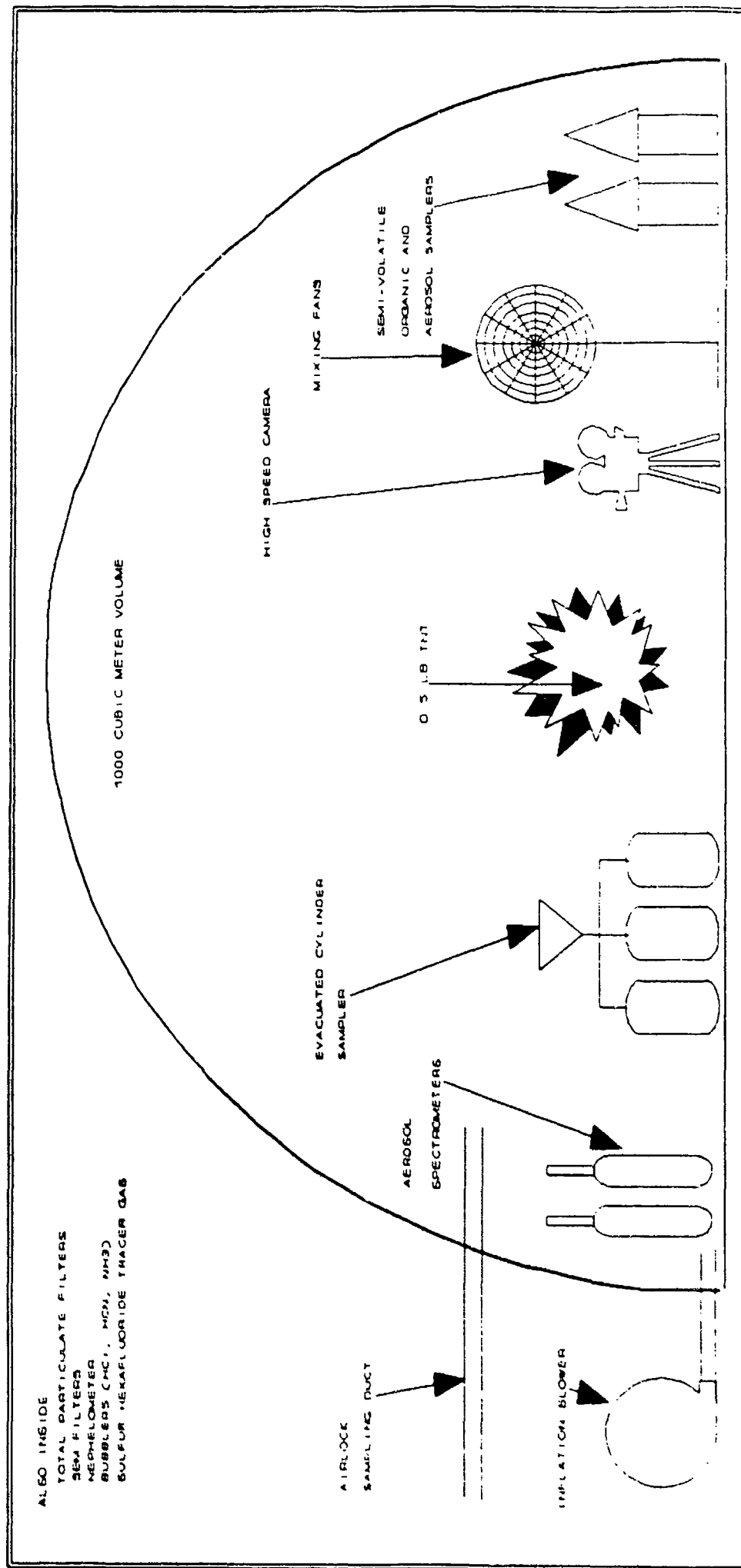


Figure 1.2 SNL Bangbox Interior OB/OD Sampling Instrumentation and Equipment.

1.4.4.9. Instrument, Sampler, and Collector Matrix. Use of instruments, samplers, and collectors, by subtest, is shown in Tables 1.2.a and b.

Table 1.2a Matrix of Instrument, Sampler, and Collector Used During the OB/OD BangBox Test.

Trial Number ^a										
Item	1	2	3	4	5	6	7	8	9	10
PARTICLE SIZERS										
DMPS ^b	X	X	X		X	X	X	X		
APS ^c	X		X	X		X	X	X	X	X
FILTERS										
Teflon ^d			X	X			X	X		X
Nuclepore ^e			X	X			X	X		X
XAD-2 ^f			X	X	X	X	X			X
Porapak-R ^g			X							
Quartz-fiber			X	X	X	X	X	X		X
DETECTORS										
Nephelometer			X	X		X	X	X	X	X
FID ^d			X					X		
PID ^e	X		X	X		X	X	X	X	X
ANALYZERS										
CO ₂	X	X	X	X		X	X	X	X	X
CO	X	X	X	X		X	X	X	X	X
SO ₂	X	X	X	X		X	X	X	X	X
O ₃	X	X	X	X		X	X	X	X	X
NO _x	X	X	X	X		X	X	X	X	X
SF ₆	X	X	X	X		X	X	X	X	X
BUBBLERS										
HCN			X	X		X	X			X
NH ₃			X	X		X	X			X
HCl										X
BAG										
1.5-m ³	X		X	X			X	X		X

Note: Footnotes identified on next page.

Table 1.2b Matrix of Instrument, Sampler, and Collector Used During the OB/OD BangBox Test.

Trial Number ^a										
Item	1	2	3	4	5	6	7	8	9	10
TANKS/CANISTERS										
0.85-L	X	X	X	X			X		X	X
6-L	X		X	X			X	X	X	X
32-L	X		X	X					X	
CAMERAS										
Color Video	X	X	X	X		X	X	X		X
HS ^c							X	X		X
HS Film	X		X							
MISCELLANEOUS										
Thermometer	X	X	X	X	X	X	X	X	X	X
Barometer	X	X	X	X		X	X	X	X	X
DP ^e Gauge	X	X	X	X		X	X	X	X	

^aTrial: 1 - Homogeneity and BB chamber volume (1 Dec 88).

2 - Ventilation rate (5 Dec 88).

3 - Single-charge TNT OD (7 Dec 88).

4 - Single-charge TNT ODs (31 Jan 89, 2 and 6 Feb 89).

5 - Extended background Air Sampling (7 Feb 89).

6 - Multiple detonation (8 Feb 89).

7 - Double-base propellant OB (9 Feb 89).

8 - Foam-attenuated TNT detonation (13 Feb 89).

9 - Multiple tank sampling ("Big Gulp") OD Trial (15 Feb 89)

10 - Composite propellant burn (16 Feb 89).

^bDifferential mobility particle sizer.

^cAerodynamic particle sizer.

^dFlame ionization detector.

^ePhotoionization detector.

^fHigh-speed.

^gDifferential pressure.

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SECTION 2. HOMOGENEITY AND BANGBOX VOLUME

2.1. Objectives

2.1.1. To establish the mixing time required to achieve homogeneity of a cloud produced by the release of SF_6 (trace gas) within the BB.

2.1.2. To establish the BB volume from the analysis of the SF_6 concentrations determined during the homogeneous phase.

2.2. Test Procedure

2.2.1. Key Data Required

2.2.1.1. The amount of SF_6 tracer released into the BB (determined from the volume and initial concentration of SF_6 tracer gas released).

2.2.1.2. The change in concentration of SF_6 in the chamber from time of release to approximately 30 min after release (sampled at 2-min intervals).

2.2.1.3. BB temperature and atmospheric pressure.

2.2.1.4. BB volume estimated from the calculations using SF_6 concentration data.

2.2.2. Data Acquisition Procedures.

The data collected during the 1 December 1988 homogeneity trial, along with data from other trials were used to assess chamber homogeneity and calculate chamber volumes.

2.2.2.1. Instrument and Facility Preparation.

2.2.2.1.1 Instruments, samplers, and collectors used are listed in Table 1.2.a and b

2.2.2.1.2 Instrument preparation. Real-time instruments were turned on at t-24 h to allow sufficient time for warm-up, stabilization, and calibration.

2.2.2.1.3 Chamber integrity. Visible small holes and tears in the BB fabric were patched with tape.

2.2.2.1.4 Ambient atmosphere. CO₂ levels were reduced to near outside, ambient levels by limiting access to the test chamber to critical personnel starting at t-45 min. From approximately t-21 to t-6 min, chamber air was flushed by directing maximum output of the inflation blower into the chamber and opening both chamber and airlock doors.

2.2.2.1.5 Pressure differential. The blower damper was manually adjusted to achieve an initial pressure differential of 18 mm (0.7 in) of H₂O between the chamber and outside atmosphere. During the trial, the pressure differential was monitored and the blower damper manually adjusted to achieve as constant a pressure differential as possible.

2.2.2.1.6 Tracer. A 0.85-L canister of SF₆ tracer gas in air was placed on the floor 2 m from the chamber wall and directly opposite the entry door. When an HS camera was used to document a trial, an electric valve on the canister was connected to the camera relay so that the canister valve would open and release SF₆ into the chamber when the HS camera was started. In all other instances, the canister valve was activated remotely.

2.2.2.1.7 Tank samplers. Three groups of three evacuated 32-L tanks were set on the floor approximately 3 m from the chamber wall, to the right of the access door.

2.2.2.1.8 Each 3-tank system (referred to as a "troika") consisted of a 20- x 25-cm quartz-fiber filter followed by a manifold connected to three 32-L tanks. Each of the three tanks had been evacuated and fitted with a remotely controlled, air-operated valve. Each system was set up so that all three tanks opened at the same time, thus drawing a total sample of 96 L (at ambient conditions) through a single quartz-fiber filter and into the tanks. Since these tanks were used for other purposes not related to calculations of BB volume or homogeneity, their use will not be discussed further here.

2.2.2.1.9 The HS camera, located 1 m from the chamber wall and opposite the chamber access door, was adjusted to take photographs at 5000 frames/s. Two stands, each containing high-intensity light bulbs, provided illumination for HS photography. Each stand was located 2 m from the explosive charge - one to the right of the charge, the other to the left.

2.2.2.1.10 Explosive. A 227gram (0.5-lb) TNT block was brought into the chamber and suspended in the charge frame approximately 1 m above the floor center at t-30 min.

2.2.2.2. Execution.

2.2.2.2.1 General comments. SNL's previous uses of the BB had not required the determination of the ventilation (leak) rate or the amount of mixing needed to establish homogeneity of the chamber atmosphere. Thus, a decision was made to release a known amount of SF_6 into the BB with mixing fans off (nonhomogeneous period). A sample from the nonhomogeneous period was taken and the fans were then turned on to create a mixed SF_6 cloud (homogeneous period). A sample from a nonhomogeneous period and a sample from a homogeneous period were required to validate a key program assumption - that the ratio of the concentration of a species to the total concentration of carbon (above ambient) in any given volume segment did not vary with time and space, regardless of the degree of homogeneity.

2.2.2.2.2 Release of approximately 10 L of SF_6 tracer gas in air at an initial concentration of approximately 500 ppmV occurred simultaneously with starting of the HS camera (if used). In all other instances, the canister valve was activated remotely.

2.2.2.2.3 Mixing (to establish a homogeneous aerosol/vapor chamber atmosphere) was accomplished in the BB with two mixing fans placed on opposite sides of the chamber, approximately 1 m from the wall, canted upwards at a 45° angle. The inflation blower exhaust was directed from a duct along the base of the interior wall, thus also contributing to mixing in the lower part of the BB. On-site discussion resulted in a decision to limit mixing time to a minimum, because the blade could act as an impingement sampler for particulate material from the detonation.

2.2.2.2.4 Approximately 2 min after release, and every 2 min thereafter, an air sample containing the SF₆ tracer was drawn from two locations within the BB; one sample from 0.7 m above the floor and the second sample 1 m from the top of the BB. Each sample was pumped into a separate 0.85-L evacuated canister until canister pressure reached 15 psi. Each canister took approximately 30 s to fill.

2.2.3. Analytical Procedures

2.2.3.1. The 0.85-L evacuated SSCs were used to sample the interior atmosphere containing SF₆ tracer gas. The SSCs are evacuated to <50 mtorr prior to sampling and then pressurized to approximately 103 kPa (15 psi) with sampled air during test. All 0.85-L canisters were assigned QA numbers and subjected to gas chromatography (GC) with an ECD assay on-site or at OGC.

2.2.3.2. Initial SF₆ assay results, available within 24 h of 1 December 1988 homogeneity trial completion, were derived from the use of an on-site Shimadzu GC with ECD. The SF₆ samples taken during trials after 1 December 1988 were assayed at OGC with the same Shimadzu GC/ECD instrument.

2.2.3.3. The degree of homogeneity was evaluated by comparing (Student "t" test) the SF₆ concentration results from the two sampler locations, together with data on the time duration that the mixing fans were operated.

2.3. Test Findings

2.3.1. Homogeneity

2.3.1.1. The first SF₆ trial was conducted on 1 December 1988. Trials, except for the multiple-detonation trials on 8 February 1989 and the TNT trial on 13 February 1989 (no SF₆ release), provided useful SF₆ analysis information from which to assess homogeneity.

2.3.1.2. Table 2.1 shows the SF₆ concentration data from the 1 December 1988 trial, together with the concentration differences between floor and ceiling sampling locations at different sampling

times. A Student "t" test was performed on the concentration differences noted between sampler locations at the 12.25 min through 32.08 min sampling times. The mean concentration differences were compared to a hypothesized mean of zero. The conclusion drawn was that the mean difference was not different from zero ($P \geq 0.95$) or stated another way the two levels of sampling indicate the chamber air is homogeneous.

2.3.1.3. The fans on the 1 December 1988 trial were turned on at $t + 9.62$ min. After reviewing the results of this trial, the on-site technical review committee decided that homogeneity within the BB should be adequately established after about 3 min of mixing. Figure 2.1 shows a plot of the SF_6 concentration data with least squares exponential fit of the data from the homogeneous period (12.25 min to the end of test).

Table 2.1 SF₆ Concentrations at the Floor and Ceiling Sampler Locations for the 1 Dec 88 Homogeneity Trial.

Event	Time ^a (min)	SF ₆ Concentration (pptv)		
		Ceiling ^b	Floor ^c	Difference ^d
Release	0.00	NA ^e	NA	NA
	2.42	4517	9121	-4604
	5.55	5055	7052	-1997
	7.67	5112	4384	728
Fans on	9.62	NA	NA	NA
	9.95	5335	4106	1229
	12.25	3774	3856	-82
	14.20	3800	3762	38
	16.23	3626	3645	-19
	18.05	3507	3534	-27
	20.00	3371	3361	10
	22.08	3304	3162	142
	24.05	3190	3076	114
	26.08	2997	3023	-26
	28.25	2992	3050	-58
	30.10	3007	2968	39
	32.08	2954	2873	81

^aTime measured from t=0 (detonation or burn time).

^bCeiling - sample intake about 1 m from top of hemisphere.

^cFloor - sample intake 0.7 m above the floor.

^dDifference in concentration between ceiling and floor sampler locations.

^eNA - not applicable.

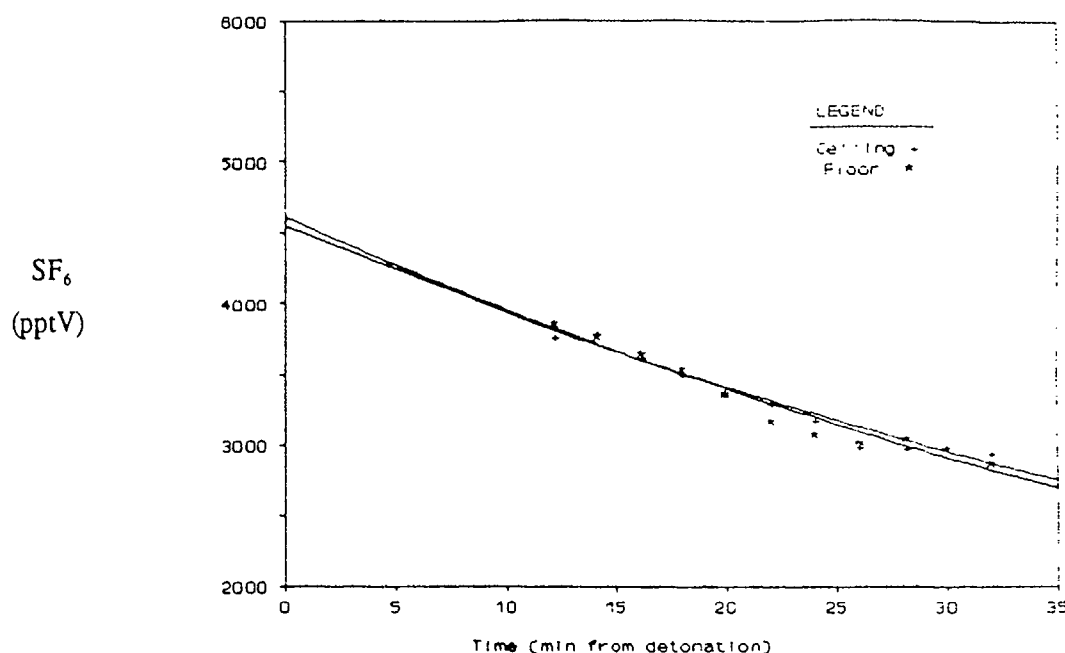


Figure 2.1 SF₆ Concentration With a Least Squares Exponential Model Fit to the Data from the 0.85-L Canister Sampler, BangBox 1 Dec 88.

2.3.1.4. The 3 min of mixing appeared to be more than adequate to achieve homogeneity on the 1 December 1988 trial; however, this homogeneity was not as well demonstrated in other trials after only 3 min of mixing. The trials on 31 January 1989 and 2 February 1989 showed the poorest agreement between floor and ceiling concentrations. This poor agreement was attributed to the malfunction of at least one mixing fan. Further investigation showed that one of the outlets in a two-outlet electrical box was defective, and both mixing fans may have been inoperative. The inflation blower did provide mixing at the lower levels, because of the air intake at the floor level and the directed airflow around the inside perimeter of the BB, but did not eliminate the concentration stratification observed on these two trials. This situation was corrected prior to testing on 6 February 1989. As noted earlier, manual adjustments were made to the inflation blower damper during each test to maintain the pressure differential needed to keep the BB properly inflated.

2.3.2. BB Volume.

2.3.2.1. The SF₆ concentration data at the ceiling and floor sampler locations were used to estimate the volume of the BB. The method of estimating this value can be stated as:

Equation 2.1 BangBox Volume

$$V_{BB} = \frac{CV_{SNL}}{C_{BB}}$$

where V_{BB} = BB volume,

C = SF₆ gas concentration in canister,

V_{SNL} = volume of gas released from canister at SNL,

C_{BB} = SF₆ concentration in BB at release.

2.3.2.2. The volume estimates for each trial and the calculated volume for a hemisphere 15.24 m (50 ft) in diameter are shown in Table 2.2. The volumes shown are adjusted to standard temperature and pressure (STP) (25°C and 760 torr). The daily average BB volume calculated from the SF₆ data was used in estimating the mass balance of carbon by the cloud volume method.

2.4. Technical Assessment

2.4.1. Mechanical mixing with fans will provide homogeneity within the BB.

2.4.2. The volume of the BB was estimated to range from 759 to 1078 m³, based on analysis of average SF₆ concentration values from sampling at two levels within the BB interior. Estimated BB volumes, calculated by applying the assumption that the BB was a hemisphere, showed similar results; but, because the volumes calculated from SF₆ data reflected the actual shape of the flexible chamber on the specific trial dates, the latter were used when a BB volume value was needed in later computations.

Table 2.2 BB Volume Calculated for Each OB/OD Trial and for a Hemisphere 15.24 m in Diameter.

Date	Location or Source	By Location ^b	Average ^c	15.24-m Diameter Hemisphere
01 DEC 88	Ceiling	1029.6		
	Floor	1126.3	1078.0	
	Calculated			831.4
31 JAN 89	Ceiling	995.0		
	Floor	672.5	833.8	
	Calculated			816.5
02 FEB 89	Ceiling	859.9		
	Floor	753.6	806.8	
	Calculated			804.9
06 FEB 89	Ceiling	808.1		
	Floor	827.3	817.7	
	Calculated			839.1
09 FEB 89	Ceiling	757.7		
	Floor	835.7	796.7	
	Calculated			846.9
15 FEB 89	Ceiling	749.5		
	Floor	768.3	758.9	
	Calculated			788.2
16 FEB 89	Ceiling	904.6		
	Floor	940.1	922.4	
	Calculated			788.2

^aAll volumes have been adjusted to standard temperature and pressure.

^bVolumes were estimated from the exponential least squares fit of the SF₆ concentration data.

^cAverage of floor and ceiling values.

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SECTION 3. BANGBOX VENTILATION RATE

3.1. Objective

To determine the BB ventilation (leak) rate under OB/OD operating conditions.

3.2. Test Procedure

3.2.1. Key Data Required

3.2.1.1. Carbon dioxide concentration monitored from detonation time ($t = \text{zero}$) to end of test.

3.2.1.2. Continuous monitoring of pressure differential between the BB and outside atmosphere.

3.2.2. Data Acquisition Procedures.

As outlined in the detailed test plan, the trial conducted 5 December 1988 was planned to serve as the only trial on which ventilation rate was to be determined; however, since data from several other trials also provided useful chamber ventilation rate values, data from all trials were used in calculations reported herein.

3.2.2.1. Instrument and Facility Preparation

3.2.2.2. Instruments and collectors used are listed in Table 1.2a and b.

3.2.2.3. Instrument preparation. All gas analyzers were allowed to run continuously following completion of the prior subtest, to allow sufficient time for warmup, stabilization, and calibration.

3.2.2.4. Chamber integrity. Visible holes and tears in the chamber fabric were patched with tape.

3.2.2.5. Atmospheric CO_2 . CO_2 levels were essentially reduced to those of the outside ambient levels by limiting access to the test chamber to critical personnel (no more than two individuals)

starting at t-45 min. Chamber air was flushed out by directing maximum output of the inflation blower into the chamber and opening both chamber and airlock doors for approximately 15 min.

3.2.2.6. Test material and tracer. A cylinder of CO_2 was positioned on the floor in the center of the chamber. A 0.85-L canister of SF_6 was placed on the floor 1 m from the chamber wall directly opposite the entry door.

3.2.2.7. Pressure differential. The blower damper was adjusted to maintain a 18 mm (0.70 in) of H_2O pressure differential between the chamber and outside atmosphere.

3.2.2.8. Data recording. Analyzer data logging instruments were turned on at t-11 min.

3.2.3. Execution

3.2.3.1. CO_2 , NO , and NO_x were sampled through direct ducting from the test chamber. Ventilation test sampling was through a tube that entered the chamber approximately 2.5 m above the floor and extended 2 m into the chamber (Figure 1.2). Air from the tube ran directly to the analyzers. The analyzers displayed CO_2 , NO , and NO_x voltage signals, digitized at 5-s intervals these data were then transmitted to the data logging instrumentation.

3.2.3.2. Operation of the inflation blower was necessary throughout the subtest to maintain the structural integrity of the BB.

3.2.4. Analytical Procedures

3.2.4.1. The real-time monitor CO_2 , NO , and NO_x concentration data, gathered during the sampling period after detonation or burn, were examined to determine which one could best be used to calculate the ventilation rate of the BB. (This rate can be determined from concentration data for any of the gaseous species that are nonreactive after their introduction into the BB. An exponential model was fit to this concentration data for the gases CO_2 , CO , NO , and NO_x , and ventilation rates were derived. The concentration data used in the least square fit were all from a period after the start of mixing. Additionally, the CO_2 data were corrected for ambient

background and worker-induced (respiration) background. An analysis of variance (ANOVA) was then performed on the derived ventilation rate values.

Equation 3.1 Exponential Model Used To Determine Ventilation Rate.

$$C_t = C_0 e^{kt}$$

where C_t = concentration of the species of interest at time = t ,
 t = time after detonation, burn, or tracer release,
 C_0 = concentration at time = 0,
 k = exponential rate of change per unit of time.

3.2.4.2. Additionally, CO_2 and CO concentration values from 6-L canister samplers provided by OGC were used in the exponential model to estimate the ventilation rate. These results were then compared with results from the real-time monitors.

3.3. Test Findings

3.3.1. After application of the least squares fit to the 140 to 288 aerosol concentration data points (from the real-time instruments) as a function of time for CO_2 , NO , and NO_x , estimates of the BB ventilation rate were derived and are shown in Table 3.1. An ANOVA indicated no statistically significant differences in the CO_2 , NO , and NO_x average ventilation rates of -0.0250, -0.0269, and -0.0243 (1/min), respectively; therefore -0.0254, the overall weighted average, was used as the best estimate of the chamber ventilation rate. This value was then used to estimate the exotic organic concentrations at t_0 based on the average concentrations of the VOST-collected samples from the BB.

Table 3.1 Ventilation Rates (k) From Least Squares Exponential Model Fit to Concentration Data from the Real-Time Instruments.

Date	CO ₂ (k/min)	NO (k/min)	NO _x (k/min)
07 DEC 88	-0.0255	ND ^a	ND
31 JAN 89	-0.0274	-0.0308	-0.0276
02 FEB 89	-0.0299	-0.0289	-0.0303
06 FEB 89	-0.0237	-0.0260	-0.0238
08 FEB 89-2 ^b	-0.0231	-0.0246	-0.0224
08 FEB 89-3	-0.0261	-0.0251	-0.0231
08 FEB 89-4	-0.0255	-0.0252	-0.0232
08 FEB 89-5	-0.0260	-0.0262	-0.0237
08 FEB 89-6	-0.0271	-0.0271	-0.0247
08 FEB 89-7	-0.0274	-0.0277	-0.0253
08 FEB 89-8	-0.0280	-0.0286	-0.0262
09 FEB 89	-0.0220	-0.0264	-0.0243
13 FEB 89	-0.0203	-0.0210	-0.0147
15 FEB 8	-0.0246	-0.0266	-0.0243
16 FEB 89	-0.0191	-0.0317	-0.0265
Mean	-0.0250	-0.0269	-0.0243
Standard Deviation	0.00296	0.00268	0.00346

^aNo data.

^bDate with detonation number; there were no CO₂, NO, and NO_x data for detonation #1.

3.3.2. As noted earlier, the 6-L canister sampler provided three or four concentration data points during the homogeneous portion of the test for CO₂ and CO. The results of the least squares exponential model fit to these concentration data are shown in Table 3.2.

Table 3.2 Ventilation Rates (k) From Least Squares Exponential Model Fit to Concentration Data from 6-L Canisters.

Date	CO ₂ (k/min)	CO (k/min)
07 DEC 88	ND ^a	ND
31 JAN 89	-0.0275	-0.0294
02 FEB 89	-0.0282	-0.0293
06 FEB 89	-0.0242	-0.0198
08 FEB 89	ND	ND
09 FEB 89	-0.0257	-0.0307
13 FEB 89	-0.0265	-0.0269
15 FEB 8	-0.0247	-0.0264
16 FEB 89	-0.0218	-0.0259
Mean	-0.0255	-0.0269
Standard Deviation	0.00217	0.00361

^a No Data

3.4. Technical Assessment

3.4.1. The BB, during homogeneous sampling periods provided a nearly constant ventilation rate. As noted above, the mean ventilation rate based on the real-time CO₂, NO, and NO_x data was -0.0254 with 95 percent confidence interval of -0.0263 to -0.0244. The mean ventilation rate based on the 6-L canister samplers for CO₂ and CO was -0.0262 with 95 percent confidence interval of -0.0279 to -0.0245. These rates are equivalent to an aerosol half-life of 27 min using the real-time concentration data, and to a half-life of 26 min using the 6-L canister data.

3.4.2. In addition to the ventilation rate, the exponential model provided estimates of the zero time (detonation or burn time) or undiluted concentration of species used in the carbon balance/emission factor calculations.

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SECTION 4. EQUIPMENT AND PROCEDURE TRIAL

4.1. Objectives

4.1.1. To determine which candidate instruments, collectors, and procedures should be used for follow on chamber trials; and

4.1.2. To pictorially document the formation and degradation of the explosion fireball.

4.2. Test Procedure

4.2.1. Data Required

4.2.1.1. Photographic coverage from t-1 to t+35 min by a video camera inside the test chamber and by a video camera outside the chamber showing the entire BB.

4.2.1.2. Motion picture coverage at 5,000 frames/s from t-0.5 to t+2 s inside the chamber by an HS camera equipped with a wide-angle lens, to document width of the fireball.

4.2.1.3. One PMS particle size distribution reading each minute (from t-45 to t+35 min) from both the ASASP 100X and forward scattering spectrometer probe (FSSP) 100X .

4.2.1.4. One DMPS particle size distribution reading every 5 min from t-45 to t+35 min.

4.2.1.5. Analog data outputs (5-s averages) from t-45 to t+35 min from the following instruments during direct sampling:

4.2.1.5.1 Nephelometer.

4.2.1.5.2 CO₂ analyzer (± 2 ppm).

4.2.1.5.3 CO analyzer (± 0.1 ppm).

4.2.1.5.4 NO_x analyzer (± 5 ppb).

4.2.1.5.5 SO₂ analyzer (± 2 ppb).

4.2.1.5.6 O₃ analyzer (± 2 ppb).

4.2.1.5.7 FID or PID for organic analyses (± 1 ppm).

4.2.1.6. Real-time test conditions:

4.2.1.6.1 Differential pressure between the interior and exterior of the BB (± 6 mm) of H₂O).

4.2.1.6.2 BB temperature ($\pm 0.5^\circ\text{C}$).

4.2.1.6.3 Bag sampler valve position.

4.2.1.6.4 Gas analyzer valve position.

4.2.1.7. Analog data outputs (5-s averages) from the following instruments on 80-L and 1.5-m³ bag air samples taken at $t-30$, $t+3$, and $t+15$ min:

4.2.1.7.1 CO₂ analyzer (± 2 ppm).

4.2.1.7.2 CO analyzer (± 0.1 ppm).

4.2.1.8. Supercritical fluid chromatography-mass spectrometry (SFC/MS) analysis of solutions of semivolatile and nonvolatile organics from:

4.2.1.8.1 Extracts of filters and resins used in the semi-VOST samplers operated inside the test chamber.

4.2.1.8.2 Extracts of filters and resins used in the semi-VOST samplers sampled from the 1.5-m³ air bag at t-30, t+3, and t+15 min.

4.2.1.8.3 Air samples collected by 32-L tanks inside the test chamber after reflux-extraction.

4.2.1.9. Gas chromatography-mass spectrometry (GC/MS) analysis of solutions of semivolatile and nonvolatile organics from:

4.2.1.9.1 Extracts of filters and resins used in the semi-VOST samplers operated inside the test chamber.

4.2.1.9.2 Extracts of filters and resins used in the semi-VOST samples collected from the 1.5-m³ air bag at t-30, t+3, and t+15 min.

4.2.1.9.3 Air samples collected by 32-L tanks inside the test chamber after reflux-extraction.

4.2.1.10. Concentrations of THC, CH₄, C₂-C₁₀HC, C₆H₆, CO, CO₂, and H₂ contained in 6-L sampling canisters operated at t-30, t+3, t+15, and t+30 min.

4.2.1.11. X-ray fluorescence (XRF) analysis (for metals) of Teflon™ filters exposed inside the BB from t-45 to t-15 min and from t+2 to t+35 min, and from Teflon™ filters exposed to the air drawn from the bag samples obtained at t-30, t+3, t+15, and t+30 min.

4.2.1.12. Scanning electron microscopy (SEM) analysis of particles from Nuclepore™ filters exposed within the test chamber from t-45 to t-15 min and from t+2 to t+35 min, and from Nuclepore™ filters exposed to the air drawn from the bag samples obtained at t-30, t+3, and t+15 min.

4.2.1.13. Concentrations of HCN and NH₃ in bubbler samples obtained from t-45 to t-15 min and from t+2 to t+35 min, using Nessler's colorimetric method for NH₃ and specific ion methods for HCN.

4.2.1.14. Concentrations of inorganic, total organic, and elemental particulate carbon from:

4.2.1.14.1 Quartz-fiber filter samples exposed within the test chamber from t-45 to t-15 min and from t+3 to t+35 min.

4.2.1.14.2 Quartz-fiber filter sample exposed to the air drawn from the bag samples obtained at t-30, t+3, and t+15 min.

4.2.1.14.3 Quartz-fiber filters samples (32-L tank systems) exposed within the test chamber at t-30, t+3, and t+15 min.

4.2.1.15. SF₆ concentrations from GC/ECD analysis of air samples, (to be performed as soon as possible after trial completion).

4.2.2. Data Acquisition Procedures

4.2.2.1. One HS camera, equipped with a wide-angle lens, was located inside the chamber, directly across the chamber from the chamber entrance. Ambient lighting provided all illumination for the HS camera.

4.2.2.2. The SF₆ release valve was electrically connected to the HS camera so that SF₆ was released at the same time the HS camera was turned on.

4.2.2.3. Semi-VOST cartridges were loaded by Alpine West Laboratories (AWL) personnel, in a SNL laboratory, one day before this subtest. Ten cartridges were each loaded with 65 g of XAD-2™ resin, and 10 cartridges were each loaded with 20 g of Porapak-R™ resin (sometimes referred to as Hayesep-R™ in test documentation). All resins were prepared by AWL at their laboratory in Provo, Utah, in accordance with AWL's letter of instruction (LOI) procedures (Volume 3), prior to the start of Chamber Test testing. Figure 4.1 shows a schematic of the semi-VOST filter and cartridges used during this subtest.

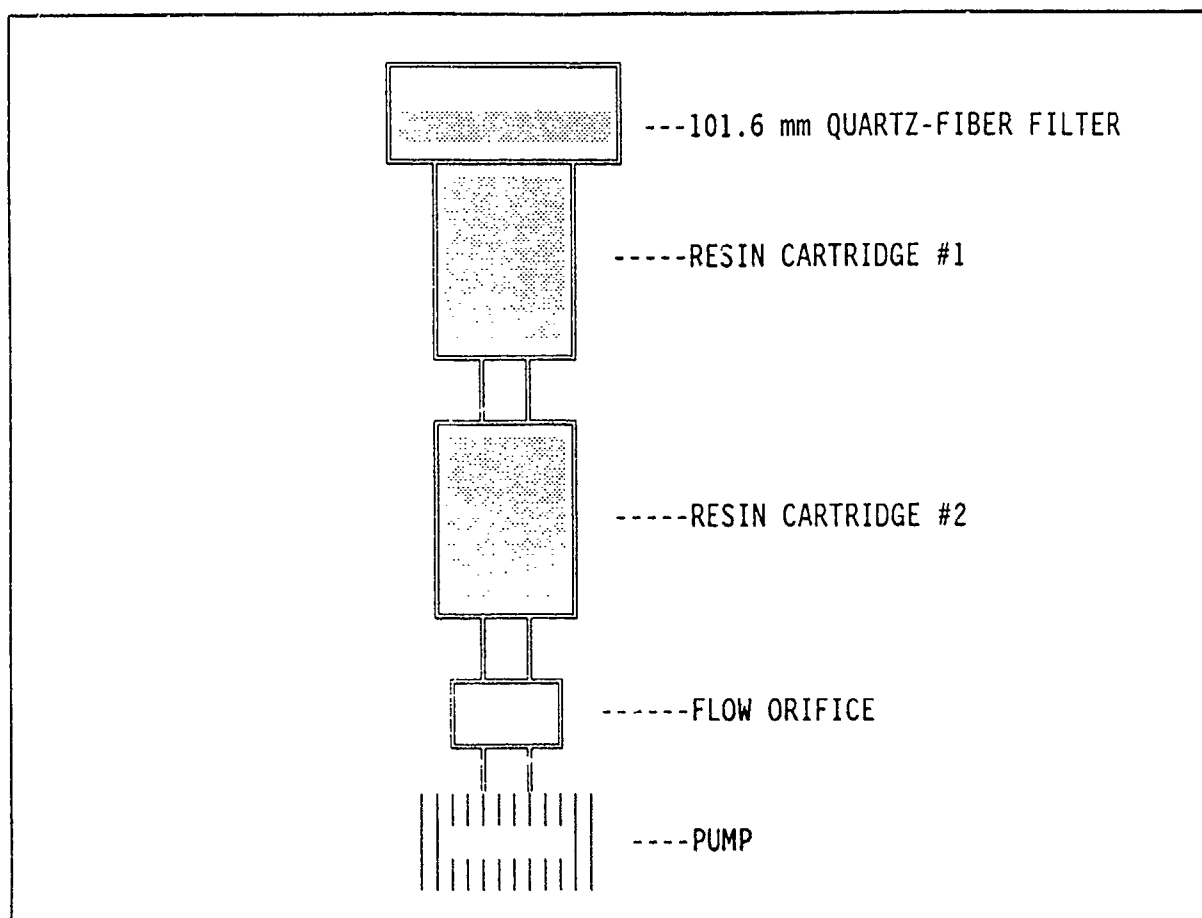


Figure 4.1 Schematic Diagram of Filter and Cartridges Used During the Equipment and Procedure Selection Trial of the BangBox Test.

4.2.2.4. The two semi-VOSTs in the chamber were designated 1 and 2; the two semi-VOSTs in the airlock were designated 3 and 4. These designations were used throughout the BB Test.

4.2.2.5. Resin-filled cartridges were inserted into the semi-VOSTs so that each semi-VOST pair had alternating leading cartridges, i.e., semi-VOSTs 1 and 3 each had a Porapak-R[™]-filled cartridge followed by an XAD-2[™]-filled cartridge; semi-VOSTs 2 and 4 each had an XAD-2[™]-filled cartridge followed by a Porapak-R[™]-filled cartridge. In all semi-VOSTs, a quartz-fiber filter preceded the two cartridges.

4.2.2.6. The NO_x analyzer was connected into the transport tube. All other real-time detectors were connected to other direct tubes to the chamber. Figure 1.1 shows a schematic of airlock sampler connections.

4.2.2.7. All real-time samplers operated continuously from the end of the previous subtest through completion of this subtest.

4.2.2.8. Background samples from outside the BB were collected using 6-L canisters sampled concurrently with a 1.5-m³ Velostat™ bag being filled with BB air.

4.2.2.9. Three 32-L tank collection systems (troikas) were located inside the chamber. Each system consisted of a 20- x 25-cm quartz-fiber filter followed by a manifold connected to three 32-L tanks. One troika of evacuated 32-L tanks functioned at t-30 min (background), a second troika at t+3 min (nonhomogeneous), and a third at t+15 min (homogeneous) using an air valve mechanism manually activated from the airlock. Each troika provided an aggregate 96-L aerosol sample.

4.2.2.10. The indirect sampling system consisted of one 1.5-m³ nominal capacity Velostat™ bag. This bag, connected to the 10-cm-diameter aluminum sampling probe that extended into the BB, was collapsed before start of sampling of the BB air and filled when a pneumatic valve was activated.

4.2.2.11. Semi-VOST, XRF, and SEM samplers drew air from the 1.5-m³ bag.

4.2.2.12. All resin-filled cartridges were individually sealed in aluminum foil immediately after removal from their semi-VOST, placed under QA control, and delivered to AWL.

4.2.3. Analytical Procedures

4.2.3.1. Quartz fiber filters and resin cartridges. Each filter and cartridge was extracted by AWL using acetonitrile. The resulting extraction fluid was divided between AWL and Battelle Columbus Division (BCD) for analysis by (SFC/MS and GC/MS, respectively) using procedures described in Volume 3.

4.2.3.2. After being placed under quality control (audit trail) and undergoing preliminary analysis by GC on-site, all 0.85-L canisters were shipped by common carrier to the OGC laboratory for analysis by GC and for archiving.

4.2.3.3. All 6-L canisters were placed under quality control (audit trail) and shipped to the OGC laboratory by common carrier for analysis by GC. Contents of 6-L canisters were analyzed by GC using procedures described in the OGC LOI in Volume 3.

4.2.3.4. All 32-L tanks were placed under quality control (audit trail) and shipped to the OGC laboratory for reflux-extraction of air samples and shipment of extracts to AWL and BCD for analysis. The organic detonation products from the aerosol, as collected by 32-L tanks, were dissolved by reflux-extraction, using methylene chloride as the solvent. The extracts were divided and equal quantities sent to AWL and BCD for analysis.

4.3. Test Findings

4.3.1. The FID hydrocarbon detector was insensitive to the OB/OD concentration levels. Use of the FID should be discontinued.

4.3.2. Use of Porapak-R™ resin in the semi-VOST appreciably reduced the air flow rate through the semi-VOST and a decision was made to use only the XAD-2™ resin.

4.3.3. Based on photographic data obtained from the HS camera film, the detonation fireball did not appear to touch the chamber wall.

4.3.4. Backpressure generated by the explosion was sufficiently strong to rupture quartz-fiber filters in the semi-VOST when they were operated in an "open face" manner.

4.3.5. Portions of resin escaped from the semi-VOST cartridge and collected in the semi-VOST cartridge container.

4.3.6. Some of the filters designated by audit-trail paperwork as "quartz-fiber filters" melted during the particulate carbon assay procedure and could not be analyzed; those that melted were glass fiber filters that had mistakenly been used.

4.3.7. At BCD, a white siloxane residue of unknown origin collected on the sides of laboratory glassware during analysis of all OGC reflux-extracted samples, except for the audit and blank samples. When AWL concentrated the samples provided by OGC, a poly-methylsiloxane gel was formed. The siloxane residue, detected during assay of reflux-extracted 32-L tank air samples, originated in grease used in the 32-L tank ball valves. The audit and blank samples, which did not produce any siloxane, came from tanks that used ungreased neoprene bellows valves.

4.3.8. Little carryover within GC columns was experienced during sample analyses. In those instances when GC carryover was suspected, it was estimated to be less than five percent.

4.4. Technical Assessment

4.4.1. The Porapak-R™ reduced airflow; therefore, XAD-2™ should be the filter resin of choice for the remainder of the trials.

4.4.2. Detonations of 227gram TNT blocks could be conducted without heat damage to the SNL chamber walls.

4.4.3. Suspected GC column carryover was so minor that it did not jeopardize results of the analysis. GC column replacement between analyses was determined to be unnecessary.

4.4.4. The resin leak from the cartridge was the result of cartridge design and will be eliminated by design modification before FWAC series tests.

4.4.5. The chemical supply house that had provided the quartz-fiber filters to AWL, had inadvertently shipped the glass fiber filter. Both filters were similar in appearance and the mistake was not detected until the filters were subjected to intense heat.

4.4.6. The 1.5-m³ Velostat™ bag should continue to be used for collecting indirect samples.

SECTION 5. TRINITROTOLUENE DETONATIONS TRIALS

5.1. Objectives

5.1.1. To provide data to permit a carbon mass balance comparison between combustion products and the mass of carbon contained in the TNT, under controlled conditions.

5.1.2. To verify the validity of measurement and sampling techniques proposed for use on the FWAC during subsequent OB/OD field tests.

5.1.3. To establish the technical suitability of SFC/MS for analysis of both aerosol and gas-phase organic samples from OB/OD trials, as compared to the more conventional GC/MS analysis method.

5.1.4. To collect, during a multiple-detonation trial (8 February 1989), using only the semi-VOST samplers in the direct sampling mode, a sufficient sample of detonation emissions to facilitate detection of very low levels of trace/exotic organic detonation products.

5.2. Test Procedure

5.2.1. Data Required.

The following data were collected during subtrials conducted on 31 January 1989, and 2, 6 8, and 15 February 1989:

5.2.1.1. Photographic video coverage from t-1 to t+35 min by a video camera inside and outside the test chamber.

5.2.1.2. Particle Size Distribution.

5.2.1.2.1 One PMS particle size distribution reading each min (from t-45 to t+35 min) from both ASASP 100X and FSSP 100X probes.

5.2.1.2.2 One DMPS particle size distribution reading every 5 min from t-45 to t+35 min.

5.2.1.3. Analog data at 5-s intervals (except during the multiple detonation trial on which data was recorded at 15-s intervals from t-45 to t+35) min from the following instruments:

5.2.1.3.1 Nephelometer.

5.2.1.3.2 CO₂ analyzer (± 2 ppm).

5.2.1.3.3 CO analyzer (± 0.1 ppm).

5.2.1.3.4 NO_x analyzer (± 5 ppb).

5.2.1.3.5 SO₂ analyzer (± 2 ppb).

5.2.1.3.6 O₃ analyzer (± 2 ppb).

5.2.1.3.7 GC-PID for organic analyses (± 1 ppm).

5.2.1.4. Data Which Indicates Test Operating Conditions.

5.2.1.4.1 BB interior/exterior (ambient) differential pressure (± 6 mm of H₂O).

5.2.1.4.2 BB temperature ($\pm 0.5^\circ\text{C}$).

5.2.1.4.3 Bag sampler valve position.

5.2.1.4.4 Gas analyzer valve position.

5.2.1.5. Analog data (5-s intervals) from the following instruments on indirect (bag) samples taken during detonation trials at t-30, t+3, and t+15 min (or later, if needed to ensure the homogeneity of the detonation products within the BB):

5.2.1.5.1 CO₂ analyzer (± 2 ppm).

5.2.1.5.2 CO analyzer (± 0.01 ppm).

5.2.1.6. Concentrations of semivolatile organics from concentrated extract solutions derived from:

5.2.1.6.1 SFC/MS analysis of each component (filters and resins) of the semi-VOST samplers operated inside the test chamber.

5.2.1.6.2 SFC/MS analysis of 32-L tank extract.

5.2.1.6.3 SFC/MS analysis of each component (filters and resins) of the semi-VOST samplers sampled from the 1.5-m³ air bag.

5.2.1.6.4 GC/MS analysis of each component (filters and resins) of the semi-VOST samplers operated inside the test chamber.

5.2.1.6.5 GC/MS analysis of components (filters and resins) of the semi-VOST samples collected from the 1.5-m³ air bag.

5.2.1.6.6 GC/MS analysis of 32-L tank extract.

5.2.1.7. Concentrations of THC, CH₄, C₂-C₁₀HC, C₆H₆, CO, CO₂, and H₂ contained in 6-L sampling canisters operated at t-30, t+3, t+15, and t+30 min.

5.2.1.8. Quantitative XRF elemental analysis of Teflon™ filters exposed inside the BB from t-45 to t-15 min and from t+2 to t+35 min.

5.2.1.9. Quantitative XRF elemental analysis of Teflon™ filter samples exposed to the air drawn from the bag samples obtained at t-30, t+3, t+15, and t+30 min.

5.2.1.10. SEM analysis results from study of particulate material from Nuclepore™ filters exposed within the test chamber from t-45 to t-15 min and from t+2 to t+35 min.

5.2.1.11. SEM analysis results from study of particulate material from Nuclepore™ filters exposed to the air drawn from the bag samples obtained at t-30, t+3, and t+15 min.

5.2.1.12. HCN and NH₃ concentrations in bubbler samples obtained from t-45 to t-15 min and from t+2 to t+35 min, using Nessler's colorimetric method for NH₃ and specific ion methods for HCN.

5.2.1.13. Volatilization and combustion analysis for volatile and elemental carbon from the quartz-fiber filters exposed within the test chamber from t-45 to t-15 min and from t+3 to t+35 min.

5.2.1.14. Volatilization and combustion analysis for volatile and elemental carbon from the quartz-fiber filters exposed to the air drawn from the bag samples obtained at t-30, t+3, and t+15 min.

5.2.1.15. Volatilization and combustion analysis for volatile and elemental carbon from the quartz-fiber filters from the 32-L tank system exposed within the test chamber at t-30, t+3, and t+15 min.

5.2.1.16. SF₆ concentrations from the GC/ECD analysis of the 0.85 L canister samplers taken as often as possible during the t-45 to t+35 min period of the trial.

5.2.2. Data Acquisition Procedures

5.2.2.1. Indirect sampling was conducted only during the 31 January and 2 and 6, February trials.

5.2.2.2. The NO_x analyzer was disconnected from the transportation tube and connected to an individual direct sampling tube.

5.2.2.3. The real-time samplers were operational from start of background to the end of trial. Sampling results were collected on data loggers and then reduced to engineering units by SNL.

5.2.2.4. A redesigned semi-VOST cartridge (Figure 5.1) was used during all BB trials on and after 31 January 1989. The lead cartridge contained 65 g of XAD-2™ resin and the trailing cartridge contained 20 g of XAD-2™ resin.

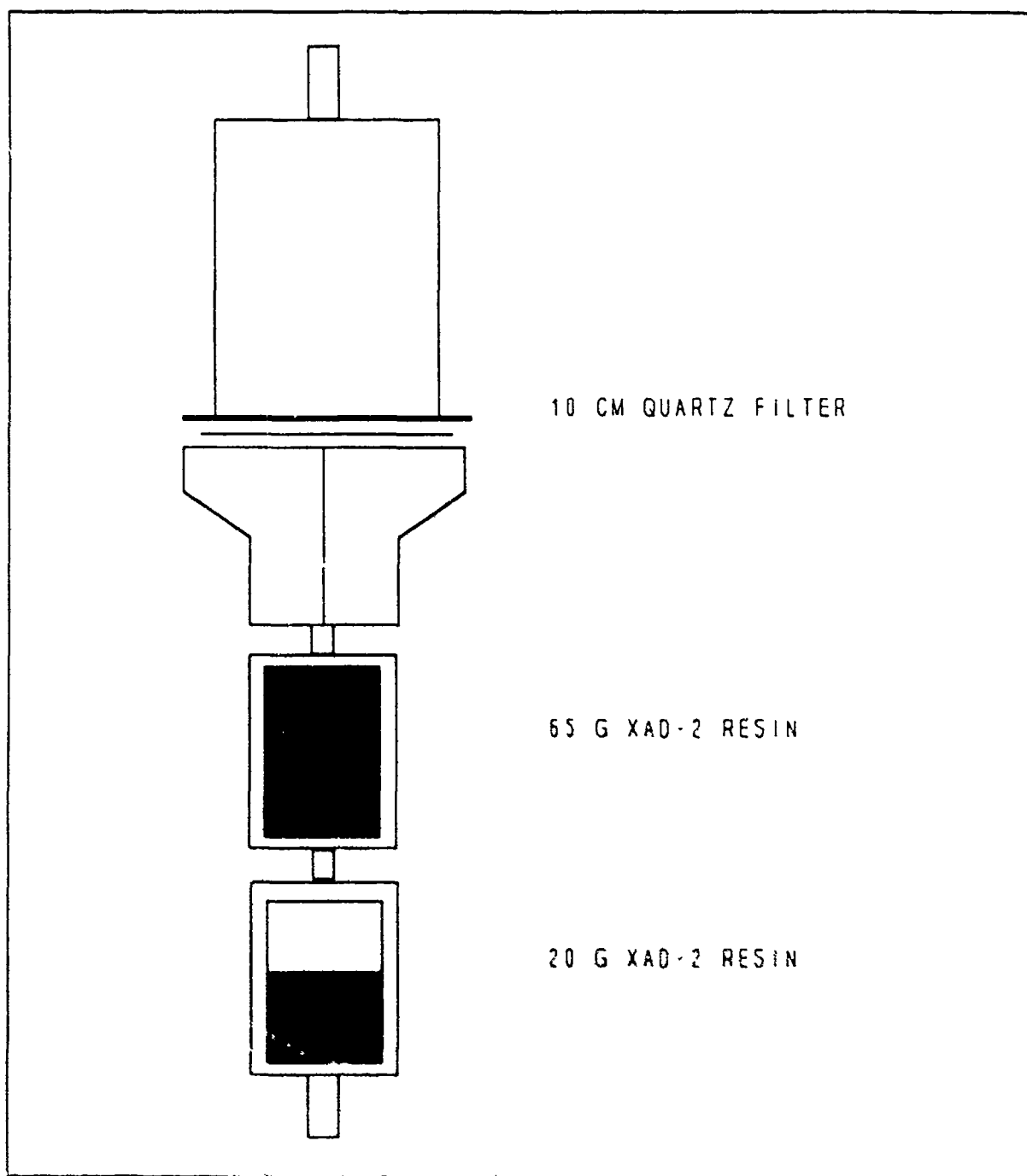


Figure 5.1 Redesigned semi-VOST Cartridge Used During OB/OD Bar/Box Test.

5.2.2.5. Sampling Systems

5.2.2.5.1 Indirect. During the 31 January 1989, and 2 and 6 February 1989 trials, the 1.5-m³ Velostat™ bag system collected the chamber air through a 10-cm diameter aluminum sampling probe which extended into the chamber. The bag was filled, and the collected aerosol was pumped through two semi-VOSTs, one Teflon™ filter, one Nuclepore™ filter, and real-time monitors during both non-homogeneous and homogeneous conditions. Procedures for operating this system are found in Volume 3.

5.2.2.5.2 Direct. Real-time instruments, bubblers, and chamber-cited semi-VOST samplers were set up and operated as previously described.

5.2.2.5.3 Tanks. Nine 32-L tank sampling systems were used during the 15 February 1989 trial. Each system, fundamentally the same as those used in previous trials, consisted of a group of three 32-L tanks, connected by a manifold, which drew their samples through a single quartz fiber filter. These 3-tank groupings were arranged randomly within the half of the test chamber to the right of the chamber entrance. One sampling group was set in Dewar flasks filled with liquid nitrogen, to condense and capture the equivalent of 10 atmospheres of sample. The nitrogen-cooled tanks were sealed after collecting the appropriate air/particulate sample. When ambient temperature was reached, each of these three tanks had an internal pressure approximately 91,400 kg/m² above atmospheric level (130 PSIG). The remaining non-cooled six tanks, sampled air until the tanks reached ambient air pressure, and then they were sealed. The organic detonation products from the air samples were then extracted by reflux-extraction of the interior of the tanks with methylene chloride.

5.2.2.5.4 Canisters. Six-L canister were used to obtain samples directly from the BB and from the 1.5-m³ Velostat™ bag. The sample was withdrawn and analyzed according to procedures found in Volume 3. SF₆ samples were drawn by 0.85 L canisters from the ceiling and 0.7 m above the chamber floor at 2-min intervals so as to obtain paired samples.

5.2.2.5.5 Two high volume direct sampling systems located within the BB consisted of two semi-VOST. This system sampled the background and, after replacing all filters, the aerosol produced

by the TNT detonation. Procedures for operating this system are found in Volume 3.

5.2.2.5.6 Particle sizing of the aerosol was accomplished with the ASASP and the FSSP located within the chamber, and with the DMPS sampling systems through a probe extending into the chamber. The operation and data output of these particle sizers are described in Volume 3.

5.2.2.5.7 Photographic coverage of the single-detonation trials (except for the 15 February trial which was not photographed) was accomplished by standard video which was operated from t-30 to t+45 min. Both test chamber interior and BB exterior were covered. Neither HS video nor HS motion picture coverage were required. Procedures used in operating the video camera were consistent with SNL procedures used in other BB demolition testing and throughout the OB/OD Chamber Test and are described in Volume 3.

5.2.2.5.8 Pressure Differential. The blower damper was manually adjusted to achieve an initial pressure differential of 18 mm (0.70 in) of H₂O between the chamber and outside atmosphere. During the trial, the pressure differential was monitored and the blower damper manually adjusted to achieve as constant a pressure differential as possible.

5.2.2.5.9 At the request of the Program Manager, an audit of procedures, equipment, and documentation was conducted by the U.S. Environmental Protection Agency (EPA), Atmospheric Research and Exposure Assessment Laboratory (AREAL), Research Triangle Park (RTP), North Carolina. Three EPA scientists conducted this audit on 6 February 1989. Letter Reports of this audit can be found in Volume 3.

5.2.3. Analytical Procedures

5.2.3.1. Sample Distribution.

5.2.3.1.1 Semi-VOST cartridges. After removal from their semi-VOST, the cartridges were sealed, identified with a QA control number, and dispatched, for assay, to laboratories for extraction and assay. To ensure prompt, undamaged delivery, and to avoid loss of analyte all samples were hand-carried by courier to assay laboratories. If the cartridges could not be transported within 24 h of

collection, they were stored at temperatures approximating -20°C until they were released to the courier.

5.2.3.1.2 Air samples. All 32-L tanks, and 6-L and 0.85 L canisters were packaged in shock-resistant containers and shipped, via common carrier, to OGC for assay.

5.2.3.1.3 Other samples. Remaining samples were packaged in sealed containers and delivered to proper laboratories for assay.

5.2.3.2. Reflux-Extraction.

5.2.3.2.1 A new reflux-extraction technique, developed at OGC, was used to extract 32-L tank air samples. The apparatus, which was designed and fabricated during the procedure development process, is shown in Figure 5.2.

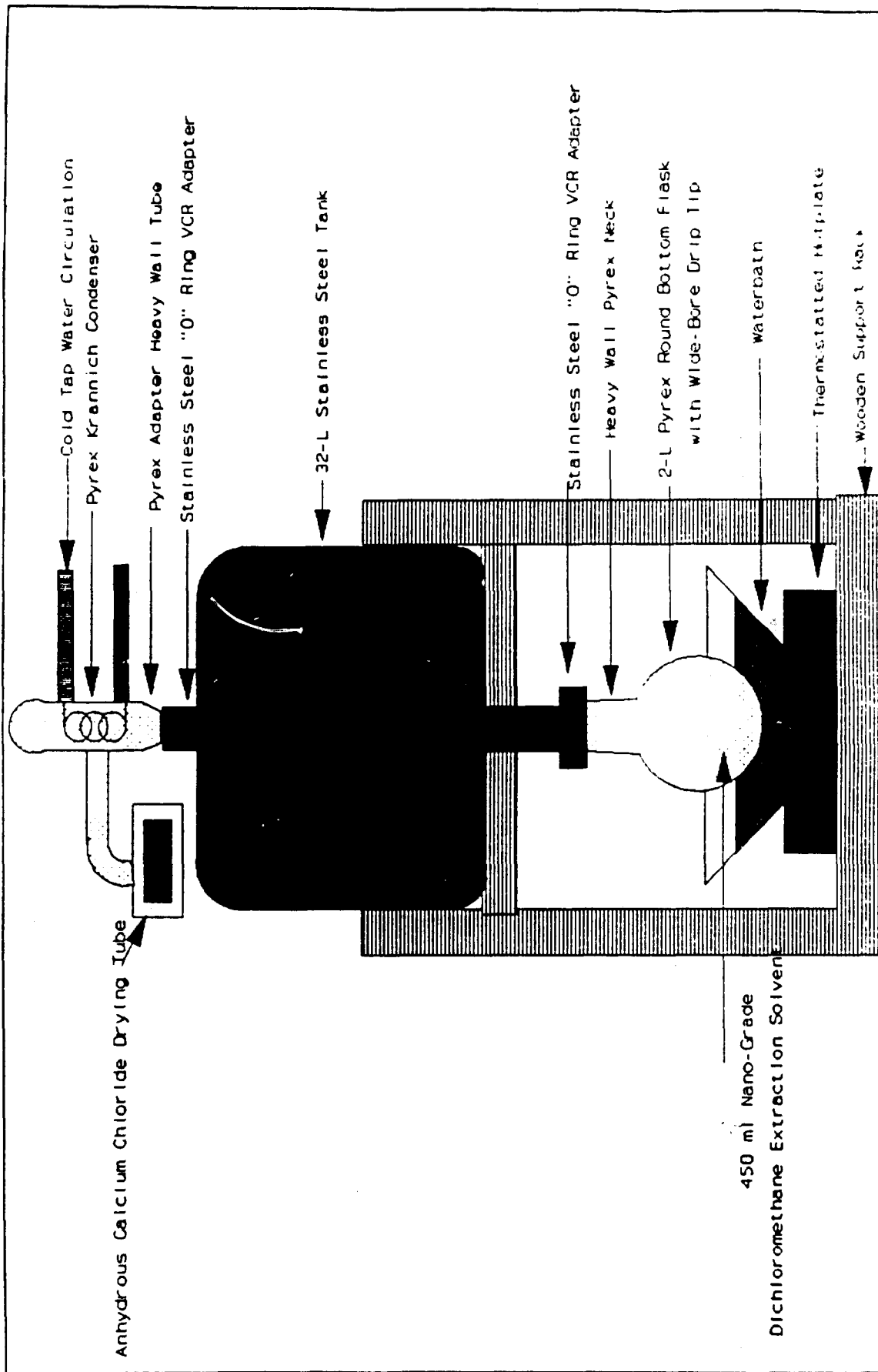


Figure 5.2 Apparatus Developed to Reflux-Extract Air Samples from 32-L Tank Samples.

5.2.3.2.2 The OGC equally divided the product of its reflux-extraction of 32-L tanks between AWL and BCD. There was no splitting or exchange of extracts from resin filters or quartz fiber filters between AWL and BCD once each laboratory had received its original samples for assay.

5.2.3.3. Detection and Identification.

5.2.3.3.1 Organics. The GC/MS was used to detect and identify gaseous and volatile organic species. Both SFC/MS and GC/MS were used to detect and identify the semivolatile organic species. Both methods were used since the standard GC/MS method could not identify and quantitate some of the target analytes. SFC/MS procedures used in this analysis are outlined in Volume 3; GC/MS procedures are found in Volume 3.

5.2.3.3.2 Elemental. XRF was used to determine elemental content. Procedures are outlined in Volume 3.

5.2.3.3.3 Carbon. Pyrolysis/combustion techniques, defined in Volume 3, were used during carbon analysis.

5.2.3.3.4 Total Suspended Particles (TSP). TSP were determined by gravimetric analysis.

5.2.3.3.5 Comparison of concentration times cloud volume method and carbon balance method. The results of the sample analyses, together with the real-time and near-real-time data, were interpreted using both the cloud volume method (considering the volume of the chamber to be the cloud volume) and the carbon balance method. Emission factors of the cloud volume method were compared to emission factors of the carbon balance method. This comparison was conducted to determine whether, on subsequent field tests, the carbon balance method could be used to determine total combustion product yield.

5.2.3.3.6 Assessment of Measurement and Sampling Techniques. Using data developed both on-site and during chemical assay, the measurement and sampling techniques used during this test were examined for their application to anticipated outdoor testing, mounted either aboard an aircraft (such as the SNL Twin Otter aircraft) or in a fixed ground location (such as in a closed laboratory

similar to, but larger than, the BB).

5.2.3.3.7 Evaluation of the SFC/MS. SFC/MS results were examined by the PM and the TSC to determine if the SFC/MS analysis method was suitable for use as the principal separation and analysis technique during future OB/OD field trials. This examination included comparison of SFC/MS results with compounds and amounts known to be in EPA-spiked samples, and, where possible, with results of the GC/MS.

5.2.3.3.8 QA/QC. QA/QC during this subtest consisted of reviewing the QC aspects of:

- a. Chamber preparation, operation, cleanup.
- b. TNT, SF₆, CO₂, and other source preparations.
- c. Record keeping.
- d. Data analysis, and sample transport, storage, handling, archiving, and analysis.
- e. Solution evaporation (concentration), extraction efficiency determination, sample dilution, and correction factor application procedures.
- f. Completeness and adequacy of data being collected/prepared.
- g. Archiving of records, data, and samples.

5.2.3.3.9 The QA review encompassed comparison of proposed action and written procedures with the highest of quality standards outlined in the literature, by nationally or internationally-accepted standards programs, manufacturer's data sheets, and other sources of accepted or approved standards. It was designed to include determining adequacy of the provision for introducing "blank", "control", "blind", and/or "split", samples. The QA agency worked closely with the EPA RTP staff and PM on the preparation, transport, and analysis of samples and the reporting of results from the spiking program. Individual laboratories were responsible for QC in their respective laboratories.

Key elements of the EPA-conducted audit included:

- a. Measurement of flow rates of all analyzers following trial completion;
- b. Comparing analyzer accuracy with EPA in-situ analyzers using known standards;
- c. Inspecting log books and calibration records;
- d. Reviewing LOIs;
- e. Observing operation of all equipment and test procedure implementation.

5.3. Test Findings

5.3.1. Carbon Mass Balance.

The mass of carbon from a TNT detonation was estimated by two methods: (1) the amount released (calculation from the molecular formula of pure TNT ($C_7H_5N_3O_6$), and from elemental analysis of a TNT field sample by an independent laboratory) (M-H-W Laboratories, Phoenix, Arizona 85018), and (2) the amount measured (accounting for all carbon-containing products through analysis of aerosol and particulate samples taken during the trial).

5.3.1.1. The calculated theoretical amount of carbon for pure TNT is 37.01 percent.

5.3.1.2. The independent laboratory analysis of two impure TNT samples before detonation gave the following results for the elemental carbon combustion:

Sample #0043:	37.68 percent carbon
Sample #0044:	37.53 percent carbon

5.3.1.2.1 These samples were nearly white and were described as "scrapings from the corners of the pressed block". Because surface scrapings are subject to ambient air exposure during long

storage and some discoloring had occurred on some of the blocks used the laboratory-analyzed samples may not be representative of the bulk of the cast TNT block actually detonated

5.3.1.3. Analysis of the aerosol and particulate samples taken during the trial provided an estimate of the total mass of carbon based on the combined carbon masses contained in CO_2 , CO, the organic carbon, and the elemental carbon generated. The carbon mass from CO_2 was estimated from the real-time (continuously monitored) instrument sampling for CO_2 with extrapolation of the fitted exponential curve (fit to the data from the homogeneous period of sampling) to detonation time ($t = 0$). The carbon mass from CO was estimated from the results of 6-L canister samples, with extrapolation of the fitted exponential curve to detonation time. Total organic carbon and elemental carbon were estimated from thermal analysis of a 1-cm² samples taken from the quartz-fiber filter of the semi-VOST (dilution corrected to $t=0$). This latter analysis incorporates a two-step volatilization and combustion process to differentiate between the contributions from volatile organic carbon and elemental carbon. Table 5.1 shows the mass of carbon derived from each of these four sources, as well as ratios of total carbon found to that predicted. The combined results of analysis of sampled aerosol and particulate carbon sources overestimate the theoretical carbon mass by 9 percent and 7 percent, using the theoretical and the elemental analysis estimates of the carbon initially present in the TNT.

Table 5.1 Carbon Mass from TNT Detonation.

Date	Source	Mass of Carbon Measured				Total Carbon Measured	Theoretical Fuel Carbon ^c (g)	Ratio ^d	Elemental Analysis Carbon (g)	Ratio ^e
		CO ₂ (g)	CO (g)	OC ^a (g)	EC ^b (g)					
31 JAN 89	TNT	94.96	0.6688	0.4161	1.0331	97.08	82.90	1.17	84.22	1.15
2 FEB 89		82.18	0.4093	0.6495	1.5160	84.75	82.13	1.03	83.43	1.02
6 FEB 89		82.92	0.3384	0.4685	2.0492	85.78	83.22	1.03	84.54	1.01
8 FEB 89-2 ^f		90.34	0.4578	0.5150	1.5426	92.86	82.99	1.12	84.31	1.10
8 FEB 89-3		90.26	0.4578	0.5150	1.5426	92.77	83.25	1.11	84.58	1.10
8 FEB 89-4		87.43	0.4578	0.5150	1.5426	89.95	82.62	1.09	83.93	1.07
8 FEB 89-5		87.15	0.4578	0.5150	1.5426	89.66	82.71	1.08	84.03	1.07
8 FEB 89-6		87.92	0.4578	0.5150	1.5426	90.43	82.42	1.10	83.74	1.08
8 FEB 89-7		86.74	0.4578	0.5150	1.5426	89.26	82.47	1.08	83.78	1.07
8 FEB 89-8		85.86	0.4578	0.5150	1.5426	88.37	81.87	1.08	83.17	1.06
15 FEB 89		83.78	0.3813	0.4751	1.4229	86.06	81.88	1.05	83.19	1.03
Average								1.09		1.07
Standard deviation								0.04		0.04

^aOC-Organic carbon.

^bEC-Elemental carbon.

^c37.01 Percent of actual mass of TNT detonated on specific trial.

^dRatio of total carbon mass measured to theoretical carbon mass in fuel carbon.

^eRatio of total carbon mass measured to independent laboratory mass of carbon.

^fNumber following date is detonation number of multi-detonation trial.

5.3.1.4. Because the elemental analysis samples may not be representative of the bulk of the actual TNT block detonated, the theoretical percent (37.01) of carbon available from pure TNT was used in all calculations. The results clearly show that the vast majority of carbon generated from the detonation of TNT is in the form of CO_2 (97.2 percent). Minor amounts of CO (0.50 percent), carbon from semivolatile or nonvolatile organics (0.57 percent), and soot (1.71 percent) are also generated. Thus, even small blocks of 74 percent oxygen-deficient TNT are very efficiently oxidized, principally to CO_2 , upon detonation in ambient air.

5.3.2. Emission Factors and Analysis Methods.

The emission factors (EFs) were calculated by two methods. The first method is referred to as the carbon balance method, and the second method is referred to as the cloud volume method.

5.3.2.1. Explanation of Carbon Balance Technique¹

5.3.2.1.1 The carbon balance technique is based on two ideas. The first is that carbon can be used as a conservative chemical tracer for the products from a high explosive (HE) detonation, or for that matter, from an OB. These processes do not consume carbon but only change its chemical form and redistribute it in space. The second idea is that the cloud of combustion or detonation products is, to a reasonable approximation, homogeneous in relative composition. That is, although the absolute concentrations of gaseous and particulate products may vary by orders of magnitude across a cloud, their relative concentrations (the concentration ratios) are approximately the same throughout, independent of position within the cloud.

5.3.2.2. Based on these assumptions, one finds that the ratio of the concentration of any combustion or detonation product D_i in some sampling volume j to the concentration of all forms of carbon originating in the event in the same sampling volume is equal to the ratio of the average concentration of detonation product D_i in the whole cloud to the average concentration of all forms of carbon from the event in the whole cloud. This is expressed mathematically:

¹Extracted from "Measuring the Composition and Total Content of Explosively Generated Smoke Clouds". Zak, B.D., Sandia National Laboratories, Albuquerque, New Mexico, July, 1988.

Equation 5.1 Basic Assumption of Carbon Balance Method.

$$\frac{[D_{ij}]}{[C_j]} = \frac{[D_i]}{[C]}$$

Here the $[C_j]$ indicates the concentration of carbon in all forms in the j th sampling volume, and the $[C]$ indicates the concentration of carbon in all forms associated with the event averaged over the whole cloud. Next, we make use of the definition of average concentration over the cloud for both product $[D_i]$ as well as for total carbon:

Equation 5.2 Definition of Average Concentration.

$$\frac{[D_{ij}]}{[C_j]} = \frac{D_{it}/V_{cl}}{C_T/V_{cl}} = D_{it}/C_T$$

Here C_T is the total mass of all forms of carbon contained in the cloud originating in the event and V_{cl} is the cloud volume. Note that if combustion is complete, and all carbon is released to the air, then C_T is equal to the total amount of carbon in the original HE or propellant. Note that the cloud volume term then drops out of the equation. So, on the basis of measurements of the relative concentration of any detonation product D_i to the concentration of all forms of carbon from the event in some sampling volume of the cloud, one can calculate the total cloud content D_a provided one knows how much carbon was contained in the original mass detonated or burned.

Equation 5.3 Total Cloud Contents of Detonation Products.

$$D_{iT} = C_T \cdot \frac{[D_i]}{[C]}$$

An emission factor EF for the i th detonation or combustion product is defined as:

Equation 5.4 Emission Factor Based on Carbon Balance Method.

$$EF_i = \frac{D_{iT}}{M}$$

Here, as before, D_{iT} is the total mass of the i th product emitted by the event into the cloud, and M is the total mass of the HE or propellant detonated or burned. To obtain the EF from the information provided by the carbon balance technique, one need only note that the total carbon mass in the HE or propellant is given by:

Equation 5.5 Total Carbon Mass.

$$C_T = F_c \cdot M$$

Here F_c is the carbon fraction for the particular HE or propellant involved in the experiment. Substituting appropriately, one finds:

Equation 5.6 Emission Factor Based on Carbon Balance Method Using Empirical Data.

$$EF_i = \frac{[D_{ij}]}{[C_j]} \cdot \frac{F_c \cdot M}{M} = F_c \cdot \frac{[D_{ij}]}{[C_j]}$$

Note: The assumptions on which this technique is based are only approximately correct; hence, the above equation is only approximate as well. Nevertheless, in actual use it has proven to be quite satisfactory as judged by the replication of results. The chief difficulty one initially encounters when attempting to apply the technique is that it is not a simple matter to measure $[C_j]$, the concentration of all forms of carbon associated with the event in a sampling volume. The difficulty arises from the fact that the most abundant final combustion or detonation product is CO_2 , and there is a natural background of CO_2 in the atmosphere of about 340 ppmV. One finds that for small amounts of HE or combustible material, it doesn't take long for the excess CO_2 in the cloud to dilute to the point that the CO_2 concentration in the cloud is indistinguishable from background. However, with state of the art techniques, one can measure CO_2 concentrations with about ± 2 ppmV uncertainty.

5.3.2.2.1 To illustrate the sampling constraints associated with this method, we take the example of 100 lbs (45.4 kg) of pure TNT, with an equivalent atomic formula of $C_7H_5N_3O_6$. Taking into account the atomic weights of the constituents, we find that TNT is 37 percent carbon by weight. If a detonation of TNT was ideal, essentially all of the carbon would be in the form of CO_2 . Thus, 16.8 kg of carbon would combine with 44.8 kg of oxygen to produce 61.6 kg of CO_2 , or 1.4 kg moles. At STP, this amount of CO_2 occupies 31.4 m^3 . We estimate that at one minute after detonation the cloud of detonation products occupies a volume of about $10^6 m^3$. This is consistent with earlier OB/OD experience. Thus, ignoring minor temperature effects, the average concentration of CO_2 in the cloud of detonation products at 1 minute is calculated to be about 31 ppmV. Between two and three minutes, experience indicates that cloud volume will have increased an order of magnitude. Hence, the average concentration of CO_2 will have fallen to about 3 ppmV. At this concentration, the uncertainty in the difference between the average cloud concentration and the background concentration of CO_2 is almost equal to the average excess CO_2 concentration in the cloud. Thus, to use the carbon balance technique with good result on a 100-lb TNT detonation, one has between one and two minutes after the event to make the measurements. Thereafter, the uncertainty on the total cloud content of the species of concern becomes too large.

5.3.2.2.2 In practice, Sandia applies the carbon balance technique by using its deHavilland Twin Otter STOL (Short Takeoff and Landing) instrumented aircraft to sample clouds and plumes. The aircraft has a 3-inch (7.62-cm) diameter sampling probe extending above and forward of the cockpit windshield. The probe transport line enters the top of the aircraft just aft of the cockpit through a gentle S-bend. Once inside the aircraft, the transport line expands to a 4-inch (10.2 cm) manifold which runs the length of the cabin to the baggage compartment at the rear. There the manifold connects to a 4-inch fast-acting pneumatic valve which in its normal position vents the probe flow out the side of the aircraft. The valve is actuated on entering a plume or cloud of interest, and returned to its normal position on emerging. When the valve is actuated, the flow is diverted into a Velostat™ (electrically conductive) plastic bag, the volume of which is a little over a cubic meter. The bag takes approximately 5 seconds to fill when the aircraft is flying at typical sampling speeds, about 50 m/s. After the pass through the plume or cloud, the Velostat™ bag contains approximately a cubic meter of air together with gaseous and particulate products drawn from the plume or cloud. As soon as the sample is captured, other valves and pumps are actuated to draw the sample into a distribution manifold, and from there to filters, real-time gas analysis instrumentation, and also

into a stainless steel canister for later laboratory analysis by gas chromatography. The real-time instrumentation always includes carbon dioxide and carbon monoxide monitors supplemented with other gaseous and particulate monitors according to the experiment being conducted. It takes 2-5 minutes, depending upon filter pumping rates, to pump the sampling bag empty. While the contents of the sampling bag are being pumped through the filters and other instrumentation, the aircraft normally makes one or two more passes through the cloud or plume using its other real-time instrumentation for characterization. Then, using quick disconnects, the filter holders and canister are changed and the aircraft is ready for another sampling pass. For a 100-lb HE shot, it is likely that the cloud would no longer be visible by this time. For larger shots, the cloud would likely be visible for quite some time, permitting additional sampling passes.

5.3.2.3. Emission Factors by the Cloud Volume Method.

EFs were calculated for gas and particulate species as follows. The intercept calculated from the regression of the real-time data to an exponential model represents the zero-time (t_0) concentration for each gas and particulate species of interest. The zero-time values were then converted from units of mixing ratio (ppm) to mass concentration (mg/m^3) at standard conditions (760 mm Hg, 25°C) by the following expression:

Equation 5.7 Conversion of Concentration of Real Time Data to Mass Concentration at Standard Temperature and Pressure.

$$\text{mg}/\text{m}^3 = (\text{ppm} \cdot \text{MW})/24.45$$

where MW = the molecular weight of the species of interest

24.45 = the molar gas volume at STP.

Emission factors were then determined by the following equation:

Equation 5.8 Emission Factor Based on Concentration Times Volume.

$$EF_i = \frac{([D_i] \cdot V)}{M}$$

EF_i = emission factor for species i

$[D_i]$ = the concentration of a detonation species i at time zero
(mg/m³)

V = the day-specific building volume (m³)

M = the mass of explosive or propellant consumed (mg)

5.3.2.4. Analysis Method

The EFs (for each compound or group of compounds) calculated by the two methods were compared using an ANOVA for a one-way randomized block design (block was the trial component) whenever the homogeneity of variance was not in question. When the treatment component of variation was significant, ($P \leq 0.05$), indicating a difference in the means, a Duncan's multiple range (MR) test was used to determine which treatments were different. There were five trial days of TNT detonation data to consider. On the 8 and 15 February 1989 trials, indirect samples were not taken due to the purposes for which those trials were performed. As a result, the real-time (SNL-continuously monitored) gas EF values from only three trial days were used in the analysis of variance, since to do otherwise would have required *estimating* 18 additional EF values from the 8 and 15 February trials. The use of only the EF's calculated from the 31 January 1989 and 2 and 6 February 1989 trial data were used to compare the carbon balance method with the cloud volume method. The effect of blocks (trials), while recognized as a source of variation in the analysis is not discussed; however, it must be accounted for in estimating the expected mass of a compound from a future detonation. Whenever the homogeneity of variance in the EF data was in question over the eight treatments, the Student "t" test was used. The ANOVA and "t" test results are described in paragraphs below, specific for each analyte. Discussion of some treatment comparisons are addressed, however, the reader is referred to the tabular results for each analyte for a complete set of the MR tests of significance.

5.3.2.5. Continuously Monitored Gases Analyzed by SNL.

EFs calculated by application of the two methods were compared for the continuously monitored gases CO_2 , NO , NO_2 , and SO_2 , based on data from SNL. The resulting EFs, calculated by the carbon balance method and the cloud volume method, for the continuously monitored gases CO_2 , NO , NO_2 , and SO_2 , (SNL data) are shown in Tables 5.2a and b.

Table 5.2a Emission Factors For CO and NO Gases, Calculated by the Carbon Balance Method and by the Cloud Volume Method.

Date 1989	Direct		Indirect					
	Carbon Balance ^b	Cloud Volume	Nonhomogeneous ^a		Homogeneous-1		Homogeneous-2	
			Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume
CO ₂								
31 Jan	1.33	1.55	1.31	9.69×10^3	1.33	1.54	1.33	1.45
02 Feb	1.31	1.36	1.28	6.35×10^3	1.31	1.30	1.31	1.21
06 Feb	1.31	1.35	1.32	1.46	1.32	1.61	1.32	1.64
08 Feb-2 ^a	1.32	1.48	NOTE: No Bag Sampling During These Trials.					
08 Feb-3	1.32	1.47						
08 Feb-4	1.32	1.43						
08 Feb-5	1.32	1.43						
08 Feb-6	1.32	1.45						
08 Feb-7	1.32	1.43						
08 Feb-8	1.32	1.42						
15 Feb	1.32	1.39						
Average	1.32	1.43	1.30	1.02	1.32	1.48	1.32	1.43
NO								
31 Jan	9.78×10^3	1.14×10^3	7.59×10^3	5.60×10^3	8.11×10^3	9.43×10^3	7.76×10^3	8.46×10^3
02 Feb	1.01×10^3	1.04×10^3	8.85×10^3	4.41×10^3	9.00×10^3	8.90×10^3	8.41×10^3	7.74×10^3
06 Feb	9.22×10^3	9.51×10^3	7.89×10^3	8.76×10^3	7.30×10^3	8.90×10^3	7.12×10^3	8.84×10^3
08 Feb-2	1.11×10^3	1.24×10^3	NOTE: No Bag Sampling During These Trials.					
08 Feb-3	1.22×10^3	1.36×10^3						
08 Feb-4	1.29×10^3	1.41×10^3						
08 Feb-5	1.25×10^3	1.35×10^3						
08 Feb-6	1.28×10^3	1.40×10^3						
08 Feb-7	1.31×10^3	1.42×10^3						
08 Feb-8	1.35×10^3	1.46×10^3						
15 Feb-8	8.81×10^3	9.26×10^3						
Average	1.14×10^3	1.25×10^3	8.11×10^3	6.25×10^3	8.14×10^3	9.07×10^3	7.76×10^3	8.35×10^3

See Footnotes following Table 5.2b

Table 5.2b Emission Factors for NO₂ and SO₂ Gases, Calculated by the Carbon Balance Method and by the Cloud Volume Method.

Date 1989	Direct ^a		Indirect					
	Carbon Balance ^b	Cloud Volume	Nonhomogeneous ^c		Homogeneous-1		Homogeneous-2	
			Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume
NO ₂								
31 Jan	3.89 x 10 ⁻⁴	4.55 x 10 ⁻⁴	1.67 x 10 ⁻³	1.23 x 10 ⁻³	1.47 x 10 ⁻³	1.70 x 10 ⁻³	2.34 x 10 ⁻³	2.55 x 10 ⁻³
02 Feb	9.61 x 10 ⁻⁴	9.91 x 10 ⁻⁴	2.30 x 10 ⁻³	1.14 x 10 ⁻³	1.52 x 10 ⁻³	1.52 x 10 ⁻³	2.60 x 10 ⁻³	2.39 x 10 ⁻³
06 Feb	9.15 x 10 ⁻⁴	9.44 x 10 ⁻⁴	9.58 x 10 ⁻⁴	1.06 x 10 ⁻³	1.31 x 10 ⁻³	1.59 x 10 ⁻³	1.94 x 10 ⁻³	2.41 x 10 ⁻³
08 Feb-2 ^d	3.70 x 10 ⁻⁴	4.14 x 10 ⁻⁴	NOTE: No Bag Sampling On These Trials.					
08 Feb-3	4.20 x 10 ⁻⁴	4.68 x 10 ⁻⁴						
08 Feb-4	3.82 x 10 ⁻⁴	4.16 x 10 ⁻⁴						
08 Feb-5	3.51 x 10 ⁻⁴	3.81 x 10 ⁻⁴						
08 Feb-6	4.62 x 10 ⁻⁴	5.07 x 10 ⁻⁴						
08 Feb-7	6.29 x 10 ⁻⁴	6.80 x 10 ⁻⁴						
08 Feb-8	4.86 x 10 ⁻⁴	5.24 x 10 ⁻⁴						
15 Feb	8.22 x 10 ⁻⁴	8.64 x 10 ⁻⁴						
Average	5.62 x 10 ⁻⁴	6.04 x 10 ⁻⁴	1.64 x 10 ⁻³	1.15 x 10 ⁻³	1.43 x 10 ⁻³	1.60 x 10 ⁻³	2.29 x 10 ⁻³	2.45 x 10 ⁻³
SO ₂								
31 Jan	1.91 x 10 ⁻⁴	2.23 x 10 ⁻⁴	1.64 x 10 ⁻⁴	1.21 x 10 ⁻⁴	1.56 x 10 ⁻⁴	1.81 x 10 ⁻⁴	1.69 x 10 ⁻⁴	1.85 x 10 ⁻⁴
02 Feb	1.10 x 10 ⁻⁴	1.13 x 10 ⁻⁴	1.81 x 10 ⁻⁴	8.99 x 10 ⁻⁵	1.72 x 10 ⁻⁴	1.71 x 10 ⁻⁴	1.74 x 10 ⁻⁴	1.61 x 10 ⁻⁴
06 Feb	1.11 x 10 ⁻⁴	1.14 x 10 ⁻⁴	1.54 x 10 ⁻⁴	1.71 x 10 ⁻⁴	1.35 x 10 ⁻⁴	1.64 x 10 ⁻⁴	1.32 x 10 ⁻⁴	1.64 x 10 ⁻⁴
08 Feb-2	1.66 x 10 ⁻⁴	1.85 x 10 ⁻⁴	NOTE: No Bag Sampling On These Trials.					
08 Feb-3	1.75 x 10 ⁻⁴	1.95 x 10 ⁻⁴						
08 Feb-4	1.76 x 10 ⁻⁴	1.91 x 10 ⁻⁴						
08 Feb-5	1.72 x 10 ⁻⁴	1.87 x 10 ⁻⁴						
08 Feb-6	1.67 x 10 ⁻⁴	1.83 x 10 ⁻⁴						
08 Feb-7	1.71 x 10 ⁻⁴	1.85 x 10 ⁻⁴						
08 Feb-8	1.91 x 10 ⁻⁴	2.06 x 10 ⁻⁴						
15 Feb	1.24 x 10 ⁻⁴	1.30 x 10 ⁻⁴						
Average	1.59 x 10 ⁻⁴	1.74 x 10 ⁻⁴	1.66 x 10 ⁻⁴	1.27 x 10 ⁻⁴	1.54 x 10 ⁻⁴	1.72 x 10 ⁻⁴	1.59 x 10 ⁻⁴	1.70 x 10 ⁻⁴

See Footnotes on page 5-24.

Footnotes to Tables 5.2a and 5.2b.

^aDirect - a sample taken directly from BB. Indirect - a sample taken directly from the BangBox, stored in the 1.5-m³ Velostat™ bag, and then sample taken from the bag.

^bCarbon balance method; Cloud volume method - Concentration times volume.

^cNonhomogeneous - Period after detonation when the detonation products have not been uniformly mixed within the enclosed volume. Homogeneous 1 and Homogeneous 2 - Homogeneous is a period after at least 3 min of fanning when the detonation products have been uniformly mixed within the enclosed volume. The 1 refers to 1st time period sample and 2 refers to 2nd time period sample.

^dDetonation number of multiple detonation trial.

5.3.2.6. Carbon dioxide (CO₂) emission factors are shown in Table 5.2a. Because variances for each of the sampling periods were not homogeneous, "t" tests were performed for selected treatment comparisons. Comparison of the EF means from the direct sampling-carbon balance method (mean = 1.32) with direct sampling-cloud volume method (mean = 1.43) showed the EF means to be different. Comparison of the indirect/nonhomogeneous/carbon balance method EF mean (1.30) with the indirect/nonhomogeneous/cloud volume method mean (1.02) showed no statistical difference between the means. That the "t" test indicated that this latter difference in means of 0.28 is insignificant but that the prior difference in mean of 0.11 is statistically significant results from the large variance associated with the nonhomogeneous-volume method values and the small number of sample values. Table 5.3 shows the means and standard errors associated with each of the methods for each sampling period.

Table 5.3 Emission Factor Means for CO₂ From the Data Collected With the Continuously Monitored SNL Instruments.

Source of Sample	BangBox Condition	Carbon Balance Method			Cloud Volume Method		
		Number of Samples	EF Mean	Standard Error	Number of Samples	EF Mean	Standard Error
Direct		11	1.32	0.00163	11	1.43	0.0170
Indirect	nonhomogeneous	3	1.30	0.0120	3	1.02	0.240
Indirect	homogeneous-1	3	1.32	0.00577	3	1.48	0.0939
Indirect	homogeneous-2	3	1.32	0.00577	3	1.43	0.124

5.3.2.6.1 In examining these means it should be kept in mind that the theoretical maximum EF value, if all the carbon in TNT is converted to CO₂, is 1.357. A comparison of the variance between the two methods was made with the results compared to the tabular "F" value for each comparison.

The result of this test showed that, in all cases, the variance associated with the cloud volume method was larger ($P \leq 0.05$) than when EF calculations were done by the carbon balance method.

5.3.2.7. Nitric oxide (NO) EFs are shown in Table 5.2a. An ANOVA indicated that the effects of treatment and blocks are significant; therefore, differences exist between the eight EF treatment means and also between the EF trial means. The matrix showing the MR treatment comparison is shown in Table 5.4, arranged from the smallest to the largest EF. The MR test showed that the EF from the non-mixed period of air sampling (indirect/nonhomogeneous/cloud volume method) is significantly lower than all other EFs by the cloud volume method and also significantly different than the EFs calculated from analysis of data by the carbon balance method from the direct sampling. Another interesting observation from the rank order of the means is that all the EFs derived from direct sampling within the chamber are larger than the EFs calculated from data from indirect sampling.

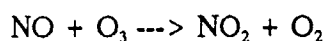
Table 5.4 Multiple Range Comparison of the Emission Factor Means for Nitric Oxide.

Sample Source*	Indirect						
	BangBox Condition	Non-homogeneous	Homo-geneous-2	Non-homogeneous	Homo-geneous-1	Homo-geneous-2	Homo-geneous-1
Indirect	Method	Cloud Volume	Carbon Balance	Carbon Balance	Carbon Balance	Carbon Balance	Cloud Volume
		Mean	6.26 x 10 ⁻³	8.11 x 10 ⁻³	7.76 x 10 ⁻³	8.14 x 10 ⁻³	9.08 x 10 ⁻³
	Non-homogeneous	Cloud Volume					
	Homo-geneous-2	Carbon Balance	Not Significant				
	Non-homogeneous						
	Homo-geneous-1						
	Homo-geneous-2	Cloud Volume	Significant at 0.05 level		Not Significant		
Homo-geneous-1					Not Significant		
Direct		Carbon Balance	Significant at 0.01 level	Significant at 0.05 level	Significant at 0.05 level	Significant at 0.05 level	Not Significant
		Cloud Volume					

^aSample - Indirect, bag; Direct from inside BangBox.

5.3.2.8. Nitrogen dioxide (NO_2) emission factors are shown in Table 5.2b. For the type of instrument used, NO_2 concentration estimates are derived by calculating the difference between the total concentration of nitrogen oxides (instrument output, NO_x) and NO . A statistical analysis was not performed however an explanation of the chemical phenomena is presented. The near-equivalence of the NO and NO_x plots of chamber concentration versus time suggests that nearly all the oxides of nitrogen produced in these detonations are in the form of nitric oxide. Any nitrogen dioxide produced most likely originates as a secondary reaction product via the reaction of ozone with nitric oxide.

5.3.2.8.1 Typical traces of NO_x , NO , and ozone voltages as functions of time following detonation are shown in Figures 5.3, 5.4, and 5.5, respectively. Pre-shot background ozone levels were typically in the range of 0.035 to 0.045 ppm. Following the detonation, NO levels rise to approximately 1 to 3 ppm and the ozone (O_3) levels drop to zero, as a result of the extremely fast reaction of NO with O_3 , as given by:



5.3.2.8.2 An upper estimate of the rate of production of NO_2 inside the chamber via this reaction pathway was calculated by determining the mass flux of ozone into the chamber (the product of ambient O_3 concentration and the inflation blower flow rate) and assuming instantaneous reaction to form NO_2 , perfect mixing inside the chamber, and no dilution effects from inflation air. Making these assumptions, the rate of change (production) of NO_2 concentration inside the chamber should be in the range of 0.001 ppm/min. Thus, after 30 minutes of reaction of detonation-generated NO and O_3 with replenishment air (from the inflation blower), no more than approximately 0.030 ppm of NO_2 would be expected to be present inside the chamber.

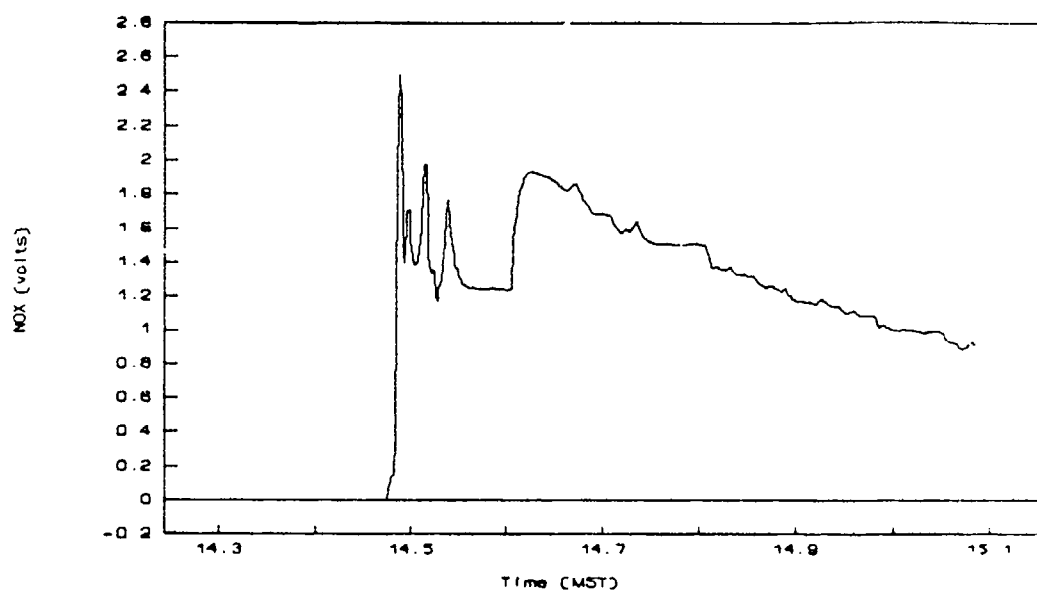


Figure 5.3 Nitrogen Oxides Voltage Data as a Function of Time, 31 Jan 89

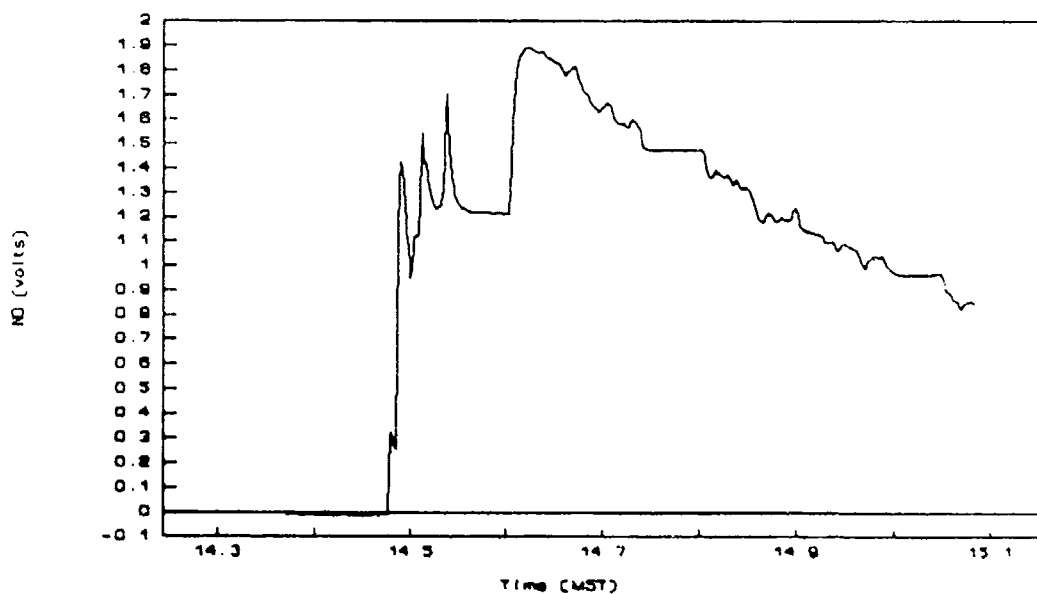


Figure 5.4 Nitric Oxide Voltage Data as a Function of Time, 31 Jan 89.

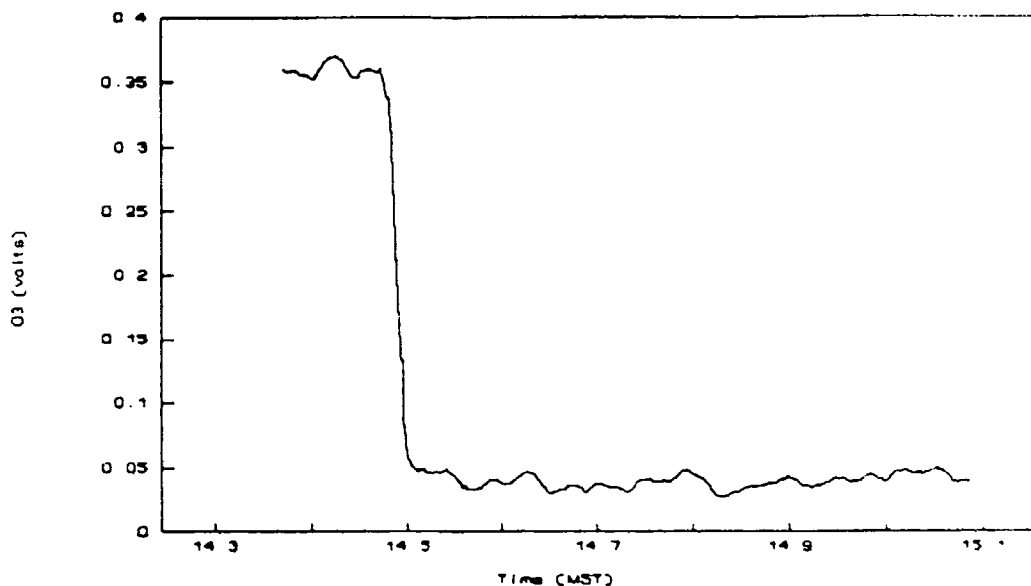


Figure 5.5 Ozone Voltage Data as a Function of Time, 31 Jan 89.

5.3.2.9. Sulfur dioxide (SO_2) emission factors are shown in Table 5.2b. The ANOVA indicated no differences between the EF means of the eight treatments and no difference between the EF means for each trial. The range of the treatment EF means was 0.000127 to 0.000174 (Table 5.5). The overall mean EF for SO_2 was 0.00015. The origin of sulfur as a trace-contaminant in the TNT is not clearly understood. It could originate from barium sulfate used as a release agent in the TNT pressing process, from the presence of residual sulfuric acid, used in the nitration process, or from the presence of residual sodium sulfite, used in TNT purification.

Table 5.5 Emission Factor Means for SO₂ from the Data Collected with the Continuously Monitored SNL Instrument.

Source of Sample	BangBox Condition	Carbon Balance Method			Cloud Volume Method		
		Number of Samples	EF Mean	Standard Error	Number of Samples	EF Mean	Standard Error
Direct		11	0.000160	9.02×10^{-6}	11	0.000174	1.12×10^{-5}
Indirect	nonhomogeneous	3	0.000166	7.88×10^{-6}	3	0.000127	2.36×10^{-5}
Indirect	homogeneous-1	3	0.000154	1.07×10^{-5}	3	0.000172	4.93×10^{-6}
Indirect	homogeneous-2	3	0.000158	1.32×10^{-5}	3	0.000170	7.55×10^{-6}

5.3.3. Volatile Gases Sampled by the 6-L Canister. The emission factors calculated by the carbon balance method for CO₂, CO, methane, acetylene, benzene, paraffins, olefins, and non-benzene aromatics, based on OGC data, are shown in Tables 5.6a through 5.6d. The emission factors for paraffins, olefins, and non-benzene aromatics, were calculated by considering the concentrations of each compound within those groups that showed an increase above background levels after detonation. The specific compounds analyzed by OGC that are included in the groupings are shown in Table 5.7. EFs for these detonation products were obtained by analyzing OGC data from 6-L canister sampling for each of the 16 treatments shown in Table 5.8. The terpene group of compounds were never detected and, thus, no EFs are given for this group.

Table 5.6a Emission Factors for the Canister-Sampled CO₂ and CO from TNT Detonations, Calculated by the Carbon Balance Method and by the Cloud Volume Method.

Date (1989)	Nonhomogeneous*		Homogeneous-1		Homogeneous-2		Homogeneous-3		Homogeneous-4	
	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume
Carbon Dioxide - Direct*										
31 Jan	1.31	0.866	1.32	1.39	1.32	1.35	1.32	1.34	1.32	1.33
2 Feb	1.28	0.630	1.31	1.20	1.31	1.18	1.31	1.14	1.31	1.13
6 Feb	1.28	0.793	1.30	1.12	1.30	1.17	1.30	1.14	1.30	1.16
15 Feb	1.29	0.739	1.31	1.17	1.31	1.18	1.31	1.18	1.31	1.19
Average	1.29	0.757	1.31	1.22	1.31	1.22	1.31	1.20	1.31	1.20
Carbon Dioxide - Indirect										
31 Jan	1.22	0.897	1.26	1.46	1.26	1.37				
2 Feb	1.19	0.591	1.24	1.23	1.26	1.16				
6 Feb	1.08	1.20	1.13	1.37	1.12	1.39				
Average	1.16	0.895	1.21	1.35	1.21	1.31				
Carbon Monoxide - Direct										
31 Jan	7.17 x 10 ³	4.75 x 10 ³	6.54 x 10 ³	6.86 x 10 ³	6.28 x 10 ³	6.43 x 10 ³	6.23 x 10 ³	6.30 x 10 ³	6.26 x 10 ³	6.29 x 10 ³
2 Feb	4.89 x 10 ³	2.41 x 10 ³	4.82 x 10 ³	4.43 x 10 ³	4.55 x 10 ³	4.09 x 10 ³	4.43 x 10 ³	3.85 x 10 ³	4.45 x 10 ³	3.85 x 10 ³
6 Feb	2.77 x 10 ³	1.71 x 10 ³	4.31 x 10 ³	3.70 x 10 ³	4.27 x 10 ³	3.83 x 10 ³	4.54 x 10 ³	3.98 x 10 ³	4.70 x 10 ³	4.17 x 10 ³
15 Feb	4.51 x 10 ³	2.57 x 10 ³	4.41 x 10 ³	3.92 x 10 ³	4.38 x 10 ³	3.92 x 10 ³	4.30 x 10 ³	3.87 x 10 ³	4.25 x 10 ³	3.85 x 10 ³
Average	4.83 x 10 ³	2.86 x 10 ³	5.02 x 10 ³	4.73 x 10 ³	4.87 x 10 ³	4.57 x 10 ³	4.87 x 10 ³	4.50 x 10 ³	4.92 x 10 ³	4.54 x 10 ³
Carbon Monoxide - Indirect										
31 Jan	5.73 x 10 ³	4.23 x 10 ³	5.46 x 10 ³	6.34 x 10 ³	5.55 x 10 ³	6.05 x 10 ³				
2 Feb	4.71 x 10 ³	2.35 x 10 ³	4.16 x 10 ³	4.12 x 10 ³	4.30 x 10 ³	3.96 x 10 ³				
6 Feb	1.21 x 10 ³	1.34 x 10 ³	2.26 x 10 ³	2.76 x 10 ³	1.55 x 10 ³	1.93 x 10 ³				
Average	3.88 x 10 ³	2.64 x 10 ³	3.96 x 10 ³	4.41 x 10 ³	3.80 x 10 ³	3.98 x 10 ³				

Table 5.6b Emission Factors for the Canister-Sampled Methane and Paraffins from TNT Detonations, Calculated by the Carbon Balance Method and by the Cloud Volume Method.

Date (1989)	Nonhomogeneous*		Homogeneous-1		Homogeneous-2		Homogeneous-3		Homogeneous-4	
	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume
Methane - Direct*										
31 Jan	2.69 x 10 ³	1.78 x 10 ³	1.48 x 10 ³	1.55 x 10 ³	1.41 x 10 ³	1.44 x 10 ³	2.10 x 10 ³	2.12 x 10 ³	4.15 x 10 ³	4.17 x 10 ³
2 Feb					Below detection level					
6 Feb	4.91 x 10 ³	3.03 x 10 ³	3.82 x 10 ³	3.28 x 10 ³	6.50 x 10 ³	5.83 x 10 ³	1.01 x 10 ⁴	8.82 x 10 ³	1.31 x 10 ⁴	1.16 x 10 ⁴
15 Feb					Below detection level					
Average	1.90 x 10 ³	1.20 x 10 ³	1.32 x 10 ³	1.21 x 10 ³	1.98 x 10 ³	1.82 x 10 ³	3.04 x 10 ³	2.74 x 10 ³	4.31 x 10 ³	3.94 x 10 ³
Methane- Indirect										
31 Jan	1.08 x 10 ³	7.97 x 10 ⁶	1.87 x 10 ³	2.17 x 10 ³	2.89 x 10 ³	3.15 x 10 ³				
2 Feb			Below detection level							
6 Feb	1.65 x 10 ³	1.83 x 10 ³	6.35 x 10 ³	7.74 x 10 ³	9.50 x 10 ³	1.18 x 10 ⁴				
Average	9.09 x 10 ⁶	8.75 x 10 ⁶	2.74 x 10 ³	3.31 x 10 ³	4.13 x 10 ³	4.99 x 10 ³				
Paraffins - Direct										
31 Jan	1.58 x 10 ³	1.05 x 10 ³	1.22 x 10 ³	1.28 x 10 ³	1.66 x 10 ³	1.70 x 10 ³	3.33 x 10 ³	3.36 x 10 ³	2.45 x 10 ³	2.46 x 10 ³
2 Feb	1.32 x 10 ³	6.50 x 10 ⁶	8.51 x 10 ⁶	7.82 x 10 ⁶	6.51 x 10 ⁶	5.85 x 10 ⁶	9.04 x 10 ⁶	7.86 x 10 ⁶	2.02 x 10 ³	1.75 x 10 ³
6 Feb	5.56 x 10 ³	3.43 x 10 ³	5.45 x 10 ³	4.69 x 10 ³	8.11 x 10 ³	7.29 x 10 ³	1.34 x 10 ⁴	1.18 x 10 ⁴	1.45 x 10 ⁴	1.29 x 10 ⁴
15 Feb	1.87 x 10 ⁶	1.07 x 10 ⁶	1.40 x 10 ⁶	1.25 x 10 ⁶	5.53 x 10 ⁷	4.96 x 10 ⁷	1.35 x 10 ⁶	1.21 x 10 ⁶	4.01 x 10 ⁶	3.63 x 10 ⁶
Average	2.16 x 10 ³	1.31 x 10 ³	1.92 x 10 ³	1.72 x 10 ³	2.62 x 10 ³	2.41 x 10 ³	4.44 x 10 ³	4.01 x 10 ³	4.84 x 10 ³	4.36 x 10 ³
Paraffins - Indirect										
31 Jan	1.10 x 10 ⁶	8.10 x 10 ³	0.00	0.00	2.94 x 10 ⁶	3.21 x 10 ⁶				
2 Feb	3.89 x 10 ³	1.93 x 10 ³	1.62 x 10 ⁶	1.50 x 10 ⁶	4.27 x 10 ⁶	3.93 x 10 ⁶				
6 Feb	1.29 x 10 ⁴	1.43 x 10 ⁴	4.23 x 10 ³	5.15 x 10 ³	7.37 x 10 ³	9.16 x 10 ³				
Average	5.64 x 10 ³	5.45 x 10 ³	1.46 x 10 ³	1.77 x 10 ³	2.70 x 10 ³	3.29 x 10 ³				

Table 5.6c Emission Factors for the Canister-Sampled Olefins and Non-Benzene Aromatics from TNT Detonations, Calculated by the Carbon Balance Method and by the Cloud Volume Method.

Date (1989)	Nonhomogeneous*		Homogeneous-1		Homogeneous-2		Homogeneous-3		Homogeneous-4	
	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume
Olefins - Direct*										
31 Jan	6.44×10^{-6}	4.26×10^{-6}	4.97×10^{-6}	5.21×10^{-6}	8.59×10^{-6}	8.80×10^{-6}	7.04×10^{-6}	7.11×10^{-6}	7.12×10^{-6}	7.15×10^{-6}
2 Feb	1.86×10^{-5}	9.18×10^{-6}	1.70×10^{-5}	1.56×10^{-5}	1.01×10^{-5}	9.05×10^{-6}	7.65×10^{-6}	6.65×10^{-6}	7.90×10^{-6}	6.84×10^{-6}
6 Feb	2.31×10^{-5}	1.43×10^{-5}	1.75×10^{-5}	1.50×10^{-5}	2.17×10^{-5}	1.95×10^{-5}	2.45×10^{-5}	2.15×10^{-5}	3.03×10^{-5}	2.69×10^{-5}
15 Feb	3.12×10^{-6}	1.78×10^{-6}	2.80×10^{-6}	2.49×10^{-6}	3.87×10^{-6}	3.47×10^{-6}	4.73×10^{-6}	4.25×10^{-6}	6.42×10^{-6}	5.81×10^{-6}
Average	1.28×10^{-5}	7.37×10^{-6}	1.06×10^{-5}	9.59×10^{-6}	1.10×10^{-5}	1.02×10^{-5}	1.10×10^{-5}	9.89×10^{-6}	1.29×10^{-5}	1.17×10^{-5}
Olefins - Indirect										
31 Jan	5.49×10^{-6}	4.05×10^{-6}	4.75×10^{-6}	5.52×10^{-6}	9.56×10^{-6}	1.04×10^{-6}				
2 Feb	3.97×10^{-6}	1.97×10^{-6}	5.40×10^{-6}	5.34×10^{-6}	1.02×10^{-5}	9.43×10^{-6}				
6 Feb	1.26×10^{-5}	1.39×10^{-5}	1.45×10^{-5}	1.77×10^{-5}	2.27×10^{-5}	2.82×10^{-5}				
Average	7.34×10^{-6}	6.65×10^{-6}	8.23×10^{-6}	9.53×10^{-6}	1.42×10^{-5}	1.60×10^{-5}				
Non-benzene Aromatics - Direct										
31 Jan	2.34×10^{-6}	1.55×10^{-6}	1.36×10^{-6}	1.42×10^{-6}	5.37×10^{-6}	5.50×10^{-6}	1.09×10^{-5}	1.10×10^{-5}	4.74×10^{-6}	4.77×10^{-6}
2 Feb	7.75×10^{-7}	3.83×10^{-7}	6.62×10^{-7}	6.08×10^{-7}	1.77×10^{-6}	1.60×10^{-6}	0.00	0.00	8.77×10^{-7}	7.59×10^{-7}
6 Feb	8.75×10^{-6}	5.39×10^{-6}	7.94×10^{-6}	6.83×10^{-6}	1.36×10^{-5}	1.22×10^{-5}	2.99×10^{-5}	2.62×10^{-5}	2.60×10^{-5}	2.31×10^{-5}
15 Feb							Below detection level			
Average	2.97×10^{-6}	1.83×10^{-6}	3.98×10^{-6}	3.58×10^{-6}	5.19×10^{-6}	4.83×10^{-6}	1.02×10^{-5}	9.31×10^{-6}	7.91×10^{-6}	7.15×10^{-6}
Non-benzene Aromatics - Indirect										
31 Jan	0.00	0.00	0.00	0.00	1.47×10^{-6}	1.60×10^{-6}				
2 Feb	8.73×10^{-6}	4.34×10^{-6}	2.16×10^{-6}	2.14×10^{-6}	4.27×10^{-6}	3.93×10^{-6}				
6 Feb	0.00	0.00	4.40×10^{-6}	5.37×10^{-6}	9.45×10^{-6}	1.17×10^{-5}				
Average	2.91×10^{-6}	1.45×10^{-6}	2.19×10^{-6}	2.50×10^{-6}	5.06×10^{-6}	5.76×10^{-6}				

Table 5.6d Emission Factors for the Canister-Sampled Acetylene and Benzene from TNT Detonations, Calculated by the Carbon Balance Method and by the Cloud Volume Method.

Date (1989)	Nonhomogeneous*		Homogeneous-1		Homogeneous-2		Homogeneous-3		Homogeneous-4	
	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume	Carbon Balance	Cloud Volume
Acetylene - Direct*										
31 Jan	5.27×10^6	5.49×10^6	4.97×10^6	5.21×10^6	4.83×10^6	4.95×10^6	5.12×10^6	5.17×10^6	5.53×10^6	5.56×10^6
2 Feb	2.32×10^6	1.15×10^6	2.36×10^6	2.17×10^6	2.37×10^6	2.13×10^6	2.09×10^6	1.81×10^6	3.51×10^6	3.04×10^6
6 Feb	9.37×10^6	5.78×10^6	8.47×10^6	7.28×10^6	1.11×10^7	1.00×10^7	1.69×10^7	1.48×10^7	1.82×10^7	1.62×10^7
15 Feb	6.24×10^7	3.56×10^7	4.66×10^7	4.15×10^7	5.53×10^7	4.96×10^7	0.00	0.00	0.00	0.00
Average	4.40×10^6	2.69×10^6	4.07×10^6	3.77×10^6	4.73×10^6	4.40×10^6	6.02×10^6	5.45×10^6	6.81×10^6	6.19×10^6
Acetylene - Indirect										
31 Jan	3.85×10^6	2.84×10^6	3.80×10^6	4.42×10^6	5.15×10^6	5.61×10^6				
2 Feb	2.38×10^6	1.18×10^6	2.16×10^6	2.14×10^6	3.41×10^6	3.14×10^6				
6 Feb	5.38×10^6	5.97×10^6	7.93×10^6	9.66×10^6	1.32×10^7	1.64×10^7				
Average	3.87×10^6	3.33×10^6	4.63×10^6	5.41×10^6	7.26×10^6	8.40×10^6				
Benzene - Direct										
31 Jan	1.76×10^6	1.16×10^6	1.36×10^6	1.42×10^6	1.07×10^6	1.10×10^6	1.28×10^6	1.29×10^6	1.58×10^6	1.59×10^6
2 Feb	2.32×10^6	1.15×10^6	3.31×10^6	3.04×10^6	1.77×10^6	1.60×10^6	3.48×10^6	3.02×10^6	2.63×10^6	2.28×10^6
6 Feb	2.50×10^6	1.54×10^6	4.24×10^6	3.64×10^6	3.72×10^6	3.34×10^6	7.66×10^6	6.73×10^6	8.67×10^6	7.70×10^6
15 Feb							Below detection level			
Average	1.64×10^6	9.63×10^5	2.23×10^6	2.03×10^6	1.64×10^6	1.51×10^6	3.11×10^6	2.76×10^6	3.22×10^6	2.89×10^6
Benzene - Indirect										
31 Jan	5.49×10^7	4.05×10^7	0.00	0.00	0.00	0.00				
2 Feb	2.38×10^6	1.18×10^6	1.62×10^6	1.60×10^6	1.71×10^6	1.57×10^6				
6 Feb	1.08×10^6	1.19×10^6	3.08×10^6	3.76×10^6	5.67×10^6	7.04×10^6				
Average	1.34×10^6	9.28×10^5	1.57×10^6	1.79×10^6	2.46×10^6	2.87×10^6				

Footnotes to Tables 5.6a through 5.6d.

^aNonhomogeneous - Period after detonation when the detonation products have not been uniformly mixed within the enclosed volume. Homogeneous is a period after at least 3 min of fanning when the detonation products have been uniformly mixed within the enclosed volume. The numbers after homogeneous refer to the sequence of the time period samples.

^bDirect - a sample taken directly from BB. Indirect - a sample taken directly from the BB, stored in the 1.5 m³ Velostat™ bag, and then a sample taken from the bag.

Table 5.7 Categories of Volatile Organic Compounds Analyzed by OGC.

Compound	Units	Compound	Units
Carbon dioxide	ppmv	Olefins - Cont'd	
Carbon monoxide		Isoprene	μg/m³
Methane		1,3-Butadiene	
Paraffins		trans-2-Pentene	
n-Heptane	μg/m³	cis-2-Butene	
2,4-Dimethylhexane		cis-2-Hexene	
2-Methylheptane		1-Pentene	
2-Methylpentane		2-Methyl-2-butene	
3-Methylpentane		1-Hexene	
Ethylcyclohexane		4-Methyl-1-pentene	
n-Hexane		trans-2-Butene	
i-Butane		i-Butene	
Methylcyclopentane		2-Methyl-2-pentene	
n-Butane		2-Methyl-1-butene	
2,4-Dimethylpentane		2-Pentene	
2,2-Dimethylpropane		Cyclopentene	
Cyclohexane		Non-Benzene Aromatics	
n-Pentane		Toluene	μg/m³
2,3-Dimethylpentane		3-Ethyltoluene	
Cyclopentane		1,3,5-Trimethylbenzene	
3-Methylhexane	n-Propylbenzene		
n-Octane	1-Ethyltoluene		
Ethane	Styrene		
2,3-Dimethylhexane	i-Propylbenzene		
Methylcyclohexane	2-Ethyltoluene		
2,3,4-Trimethylpentane	Ethylbenzene		
n-Nonane	o-Xylene		
2,3-Dimethylbutane	p-Xylene		
2,2,3-Trimethylpentane	m-Xylene		
i-Pentane	1,2,4-Trimethylbenzene & sec-Butylbenzene		
Propane			
2,2-Dimethylbutane	Acetylene	μg/m³	
3-Ethylhexane	Benzene	μg/m³	
Olefins		Terpenes	
Ethylene	μg/m³	β-Pinene	μg/m³
2-Methyl-1-pentene		α-Terpinene	
Propene		d-Limonene	
1-Butene		γ-Pinene	
trans-2-Hexene		Δ¹-Carene	
3-Methyl-1-butene		γ-Terpinene	
Myrcene		Terpinolene	

Table 5.8 Sampling Periods Which Provided Species Concentration Data for Calculating Emission Factors.

Source of Sample	Mixed Condition	Method
Direct	Nonhomogeneous	Carbon Balance
	Nonhomogeneous	Cloud Volume
	Homogeneous 1	Carbon Balance
	Homogeneous 1	Cloud Volume
	Homogeneous 2	Carbon Balance
	Homogeneous 2	Cloud Volume
	Homogeneous 3	Carbon Balance
	Homogeneous 3	Cloud Volume
	Homogeneous 4	Carbon Balance
	Homogeneous 4	Cloud Volume
Indirect	Nonhomogeneous	Carbon Balance
	Nonhomogeneous	Cloud Volume
	Homogeneous 1	Carbon Balance
	Homogeneous 1	Cloud Volume
	Homogeneous 2	Carbon Balance
	Homogeneous 2	Cloud Volume

5.3.3.1. Carbon dioxide EFs calculated from OGC 6-L canister data are shown in Table 5.6a. Because variances for each of the sampling periods were not homogeneous, "t" tests were performed for selected treatment comparisons. Comparison of the EF means from the direct sampling/nonhomogeneous/carbon balance method (mean = 1.29) with the direct sampling/nonhomogeneous/cloud volume method (mean = 0.757) showed the means to be statistically different. Comparison of the indirect sampling/nonhomogeneous/carbon balance method EF mean (1.16) with the indirect sampling/nonhomogeneous/cloud volume method EF mean (0.896) showed no statistically significant difference between the means. Table 5.9 shows the means and standard errors associated with each of the methods of calculating EFs for each sampling period. A comparison of the variances between the two methods at each of the sampling periods showed the variance, in every case, to be larger for the cloud volume method. For CO₂, the carbon balance method is associated with greater precision and EFs that are consistent throughout the homogeneous and nonhomogeneous periods of the test.

Table 5.9 Emission Factor Means for CO₂ From the Assay for Volatile Organics.

Source of Sample	Chamber Condition	Carbon Balance Method			Cloud Volume Method		
		Number of Samples	EF Mean	Standard Error	Number of Samples	EF Mean	Standard Error
Direct	Nonhomogeneous	4	1.29	0.00707	4	0.757	0.0497
	Homogeneous-1	4	1.31	0.00408	4	1.22	0.0590
	Homogeneous-2	4	1.31	0.00408	4	1.22	0.0434
	Homogeneous-3	4	1.31	0.00408	4	1.20	0.0476
	Homogeneous-4	4	1.31	0.00408	4	1.20	0.0442
Indirect	Nonhomogeneous	3	1.16	0.00426	3	0.896	0.0176
	Homogeneous-1	3	1.21	0.00404	3	1.35	0.0669
	Homogeneous-2	3	1.21	0.00467	3	1.31	0.0736

5.3.3.2. Carbon monoxide emission factors are shown in Table 5.6a. The analysis indicated that the effects of treatment and blocks are significant; therefore, differences exist between the 16 EF treatment means and the EF trial means. The matrix showing the MR treatment mean comparisons are shown in Table 5.10, and is arranged from the smallest to the largest EF. (The table is truncated after the indirect/homogeneous 2/cloud volume comparison data; however, all comparisons beyond this point showed EF mean differences not to be significant.) The EF means derived from applying the cloud volume method to data for the nonhomogeneous sampling period (0.00264 and 0.00286) were significantly lower than the EF means calculated from data for all other sampling periods. Other comparisons also show significant differences in EF means. The most interesting feature of these comparisons was that all EF means calculated from data derived from other indirect measurements ranked lower than EF means from the direct sampler measurements. EF means from the direct sampling (other than the nonhomogeneous/cloud volume method values) were not different from each other. These direct sampling EF means ranged from 0.00450 to 0.00502.

5.3.3.3. Methane EF values are shown in Table 5.6b. The analysis indicated that the effects of treatment and blocks are significant; therefore, statistically significant differences exist between the 16 EF treatment means and the EF trial means. The matrix showing the MR EF treatment mean comparisons performed is shown in Table 5.11, arranged from the smallest to the largest. (The table is truncated after the direct/homogeneous 1/carbon balance method comparisons; however, all comparisons beyond this point showed no significant differences.) As can be observed from the means in Table 5.11, the mean EF for methane produced varies from 0.00000876 to 0.0000498. Thus, by applying the mean emission factor of 0.0000498 to a one-pound TNT block detonated in the BB, one would expect a maximum of 49.8-millionths of a pound of methane to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than one tenth of a pound ((0.0996 lb) or 45.2 g) of methane would result.

5.3.3.4. Paraffin hydrocarbon EFs are shown in Table 5.6b. The analysis indicates that the effects of treatment and blocks are significant; therefore, differences exist between the 16 EF treatment means and the EF trial means. The matrix showing the MR treatment mean comparisons is in Table 5.12, arranged from smallest to largest EF means. (The table is truncated after the direct/nonhomogeneous/cloud volume method comparisons; however, all comparisons beyond that point showed no significant differences.) No statistically significant difference was noted when the EF mean for the direct/nonhomogeneous/carbon balance method calculation (0.0000216) was compared with the EF mean from the direct/nonhomogeneous/cloud volume method (0.0000131). Also, no difference was noted when the EF mean calculated by the indirect/nonhomogeneous/carbon balance method (0.0000563) was compared to the EF mean calculated by the indirect/nonhomogeneous/cloud volume method (0.0000544). The EF means in Table 5.12 vary from 0.0000131 to 0.0000563. Thus, by applying the mean EF of 0.0000563 to a one-pound TNT block detonated in the BB, one would expect a maximum of 56.3-millionths of a pound of paraffins to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than two-tenths of a pound ((0.1126 lb) or 51.1 g) of paraffins would result.

5.3.3.5. Olefin emission factors are shown in Table 5.6c. The analysis indicates that the effects of treatment and blocks are significant; therefore, differences exist between the 16 EF treatment means and EF trial means. The matrix showing the MR treatment mean comparisons are shown in Table 5.13, arranged from smallest to largest EF means. (The table is truncated after the direct/homogeneous 3/cloud volume method comparisons; however, all comparisons beyond that point are not significant.) As can be observed from the MR results, there is no difference in the EF means calculated by using any of the direct sampling values (range 0.00000738 to 0.0000129). The EF means varied from 0.00000664 to 0.0000160. Thus, by applying the mean EF of 0.0000160 to a one-pound TNT block detonated in the BB, a maximum of 16.0-millionths of a pound of olefins is expected to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than one-tenth of a pound ((0.032 lb) or 14.5 g) of olefins would result.

5.3.3.6. Nonbenzene aromatic compound emission factors are shown in Table 5.6c. The analysis indicates that the effect of blocks is significant; therefore, differences exist between the EF trial means. While differences in EF treatment means were not detected, a listing of the means is given which shows that the cloud volume method yields the smallest EFs when nonhomogeneous sampling data are involved (Table 5.14). The EF treatment means vary from 0.00000145 to 0.0000102. Thus, by applying the mean emission factor of 0.0000102 to a one-pound TNT block detonated in the BB, one would expect a maximum of 10.2-millionths of a pound of nonbenzene aromatics to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than one-tenth of a pound ((0.0204 lb) or 9.26 g) of nonbenzene aromatics would result.

Table 5.14 Multiple Range Comparison of the Emission Factor Means for Nonbenzene Aromatic Hydrocarbons.

[illegible]

*Sample - Indirect, (bag). Direct from inside BangBox chamber.

Not significant.

5.3.3.7. Acetylene EFs are shown in Table 5.6d. The analysis indicated that effects of treatment and blocks are significant; therefore, differences exist between the 16 EF treatment means and the EF trial means. The matrix showing the MR treatment means comparison is shown in Table 5.15, arranged from the smallest to the largest EF mean. (The table is truncated after indirect/homogeneous 1/carbon balance method; however, all comparisons beyond that point indicated that noted differences were not significant). The nonhomogeneous/cloud volume method of yielded the smallest EF means (0.00000269 and 0.00000333); however, these EF were not significantly different from the nonhomogeneous/carbon balance method of calculating EF means (0.00000387 and 0.00000440). Thus, by applying the mean emission factor of 0.0000083 to a one-pound TNT block detonated in the BB, one would expect a maximum of 8-millionths of a pound of acetylene to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than one-tenth of a pound ((0.0166 lb) or 7.53 g) of acetylene would result.

5.3.3.8. Benzene emission factors are shown in Table 5.6d. The analysis indicated that effects of treatment and blocks are significant; therefore, differences exist between the 16 EF treatment means and the EF trial means. The matrix showing the MR treatment means comparison is shown in Table 5.16, arranged from the smallest to the largest EF mean. (The table is truncated after direct/homogeneous 2 cloud volume method; however, all comparisons beyond that point indicated that noted differences were not significant). As can be observed from the MR, results there is no difference in the EF means (0.00000134 to 0.00000322) after removing the nonhomogeneous/cloud volume method EF means (0.000000925 and 0.000000962). By applying the largest emission factor mean of 0.00000322 to a one-pound TNT block detonated in the BB, one would expect a maximum of 3.22-millionths of a pound of benzene to be produced as a combustion product. For one-ton (2,000 lb) TNT detonation, provided linear scaling applies, less than one one-hundredth of a pound (0.00644) or 2.92 g) of benzene would result.

Table 5.16 Multiple Range Comparison of the Emission Factor Means for Benzene.

Sample Source ^a			Indirect	Direct	Indirect	Direct
BangBox Condition	Method	Mean	9.25 x 10 ⁻⁷	9.62 x 10 ⁻⁷	1.34 x 10 ⁻⁶	1.51 x 10 ⁻⁶
		Cloud Volume	9.25 x 10 ⁻⁷			
	Indirect	Nonhomogeneous	Cloud Volume	9.62 x 10 ⁻⁷	1.34 x 10 ⁻⁶	1.51 x 10 ⁻⁶
	Direct		Carbon Balance			
	Indirect		Cloud Volume	Not Sig		
	Direct	Homogeneous-2	Carbon Balance		Not Sig	Not Sig
	Indirect	Homogeneous-1	Cloud Volume			
	Direct	Homogeneous-2	Carbon Balance			
	Indirect	Nonhomogeneous	Cloud Volume			
	Direct	Homogeneous-1	Carbon Balance			
	Indirect		Cloud Volume			
	Direct		Carbon Balance			
	Indirect	Homogeneous-2	Cloud Volume			
	Direct	Homogeneous-3	Cloud Volume			
	Indirect	Homogeneous-2	Cloud Volume			
	Direct	Homogeneous-4	Cloud Volume			
	Indirect	Homogeneous-3	Carbon Balance			
	Direct	Homogeneous-4	Carbon Balance			

^aSample - Indirect, (bag), Direct from inside BangBox chamber.

^bNot significant.

^cLevel of significance.

5.3.3.9. Terpene emission factors. Of the seven terpene-like compounds listed in Table 5.7 none were detected above the lower detection limit of the GC analysis procedure used.

5.3.3.10. Semivolatile Organics.

5.3.3.10.1 The test design provided data for calculation of EFs for semivolatile organics from the direct semi-VOST, the indirect/nonhomogeneous semi-VOST, and the indirect/homogeneous semi-VOST. The net amounts of semivolatile organic compounds, calculated by summing the amounts found in each of the three components of the sampling train after correcting for background, were converted to EFs for each of the following compounds:

2,6-Dinitrotoluene
4-Nitrophenol
2,4-Dinitrotoluene
2-Nitronaphthalene
2,4,6-Trinitrotoluene
1-Nitropyrene
Phenol
Dibenzofuran
Benz[*a*]anthracene
Benzo[*a*]pyrene
Dibenz[*a,h*]anthracene
N-Nitrosodiphenylamine
Naphthalene
1- & 2-Methylnaphthalene
1,3,5-Trinitrobenzene
Biphenyl
Phenanthrene
2,5-Diphenyloxazole
1,1'-3-Trimethyl-3-phenylindane
Pyrene

5.3.3.11. The following compounds, although looked for, were never found:

1,6-Dinitropyrene

2-Naphthalamine

Major Unknown

Benz[c]acridine

5.3.3.12. The 25 target analyte organic compounds cited in the two lists above are those which were searched for and, measured by GC/MS and/or SFC/MS selected-ion monitoring techniques. Basically, the target analyte list consisted of: expected organic components that may be present in the block of TNT detonated in the chamber; combustion product compounds which, if present in high concentration, would be of environmental concern; and unexpected compounds that were detected in low concentration once analyses of actual samples were begun, and which were added to the list of target analytes monitored by SFC/MS and/or GC/MS.

5.3.3.13. The analysis results for each of the 21 compounds that were detected are discussed separately in subparagraphs below. These discussions include a value for the EF mean and its 95 percent confidence interval. In computing this mean, all pertinent analysis data are combined for the given species; thus, any variation contributed by differences in EFs calculated using data from different trial days, sampler source (direct, indirect/nonhomogeneous, and indirect/homogeneous), analytical lab, or individual instrument is included as a part of the overall EF error variation. Missing cells in the tables of calculated EF values are due to the fact that a number of the quartz-fiber filters from the semi-VOST were only assayed for organic carbon and elemental carbon; no SFC/MS or GC/MS assays were performed on these filters to yield EF values for the semivolatile organics.

5.3.3.14. The semivolatile compound observed and corrected for background (net concentration values) were used in the calculation of EF.

5.3.3.14.1 2,6-Dinitrotoluene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.17.

Table 5.17 Emission Factor Values for 2,6-Dinitrotoluene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	1.83×10^{-4}		
	2	3.27×10^{-7}	3.49×10^{-7}	2.72×10^{-7}
2 Feb 89	1		0.00	8.63×10^{-4}
	2	9.15×10^{-4}	4.39×10^{-7}	0.00
6 Feb 89	1	0.00		
	2	1.02×10^{-7}	5.96×10^{-4}	1.98×10^{-4}

The mean EF and associated 95 percent confidence interval are 1.35×10^{-7} , and 4.23×10^{-4} to 2.29×10^{-7} , respectively. The largest EF from the 13 estimates was 0.000000439 (4.39×10^{-7}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 439-billionths of a pound of 2,6-dinitrotoluene to be produced as a combustion product, or released as a contaminant of the TNT. Thus, for a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than one one-thousandth of a pound ((0.000878 lb) or 0.398 g) of 2,6-dinitrotoluene would result.

5.3.3.14.2 4-Nitrophenol. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.18.

Table 5.18 Emission Factor Values for 4-Nitrophenol From the BangBox TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	4.52×10^{-7}		
	2	0.00	0.00	0.00
2 Feb 89	1		8.34×10^{-7}	2.59×10^{-6}
	2	0.00	0.00	0.00
6 Feb 89	1	5.05×10^{-7}		
	2	0.00	0.00	0.00

The mean EF and associated 95 percent confidence interval are 3.37×10^{-7} , and 0 to 7.78×10^{-7} , respectively. The largest EF from the 13 estimates was 0.00000259 (2.59×10^{-6}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 2.59-millionths of a pound of 4-nitrophenol to be produced as a combustion product, or released as a contaminant of the TNT. Thus, for a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than six one-thousandth of a pound ((0.00518 lb) or 2.35 g) of 4-nitrophenol would result. It should be noted that laboratory 2 never found 4-nitrophenol and laboratory 1 always found 4-nitrophenol. This difference between laboratories is still unresolved. Submission of duplicate samples to a third, independent laboratory yielded inconclusive GC/MS results as to the presence of 4-nitrophenol.

5.3.3.14.3 2,4-Dinitrotoluene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.19.

Table 5.19 Emission Factor Values for 2,4-Dinitrotoluene From the BangBox TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	5.17×10^{-8}		
	2	9.43×10^{-8}	3.17×10^{-7}	1.05×10^{-6}
2 Feb 89	1		1.24×10^{-7}	1.29×10^{-7}
	2	0.00	0.00	0.00
6 Feb 89	1	7.54×10^{-8}		
	2	8.43×10^{-8}	2.54×10^{-8}	1.97×10^{-7}

The mean EF and associated 95 percent confidence interval are 1.65×10^{-7} , and 0 to 3.35×10^{-7} , respectively. The largest EF from the 13 estimates was 0.00000105 (1.05×10^{-6}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 1.05-millionths of a pound of 2,4-dinitrotoluene to be produced as a combustion product, or released as a contaminant of the TNT. Thus, for a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than one one-thousandth of a pound ((0.000210 lb) or 0.953 g) of 2,4-dinitrotoluene would result.

5.3.3.14.4 2-Nitronaphthalene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.20.

Table 5.20 Emission Factor Values for 2-Nitronaphthalene From the BangBox TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	1.51×10^{-8}		
	2	2.18×10^{-7}	5.92×10^{-7}	5.51×10^{-8}
2 Feb 89	1		0.00	8.63×10^{-8}
	2	1.18×10^{-8}	6.43×10^{-7}	1.13×10^{-8}
6 Feb 89	1	2.30×10^{-8}		
	2	2.10×10^{-7}	1.71×10^{-7}	9.87×10^{-8}

The mean EF and associated 95 percent confidence interval are 1.64×10^{-7} , and 3.42×10^{-8} to 2.24×10^{-7} , respectively. The largest EF from the 13 estimates was 0.000000643 (6.43×10^{-7}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 643-billionths of a pound of 2-nitronaphthalene to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than two one-thousandths of a pound ((0.00129 lb) or .583 g) of 2-nitronaphthalene would result.

5.3.3.14.5 2,4,6-Trinitrotoluene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.21.

Table 5.21 Emission Factor Values for 2,4,6-Trinitrotoluene From the BangBox TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	1.18×10^{-6}		
	2	1.29×10^{-8}	1.54×10^{-8}	0.00
2 Feb 89	1		3.38×10^{-6}	2.74×10^{-6}
	2	0.00	0.00	0.00
6 Feb 89	1	7.35×10^{-7}		
	2	2.56×10^{-9}	2.26×10^{-8}	7.05×10^{-9}

The mean EF and associated 95 percent confidence interval are 6.23×10^{-7} , and 0 to 1.32×10^{-6} , respectively. The largest EF from the 13 estimates was 0.00000338 (3.38×10^{-6}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 3.38-millionths of

a pound of 2,4,6-trinitrotoluene to be released unoxidized. For a one-ton (2,000 lb) TNT detonation, provided linear scaling applies, less than seven one-thousandths of a pound ((0.00676 lb) or 3.07 g) of residual 2,4,6-trinitrotoluene would result.

5.3.3.14.6 1-Nitropyrene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.22.

Table 5.22 Emission Factor Values for 1-Nitropyrene From the BangBox TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	0.00		
	2	0.00	1.06×10^{-6}	0.00
2 Feb 89	1		0.00	0.00
	2	0.00	0.00	0.00
6 Feb 89	1	0.00		
	2	0.00	0.00	0.00

Only 1 of 13 sample assays of the semi-VOST showed the presence of 1-nitropyrene. Although the value is well above the EF, based on the detection limit for 1-nitropyrene ($EF = 1.83 \times 10^{-8}$), it is still considered to be an artifact because it was not found in any other samples.

5.3.3.14.7 Phenol. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.23.

Table 5.23 Emission Factor Values for Phenol From the BangBox TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	2.84×10^{-6}		
	2			
2 Feb 89	1		2.52×10^{-5}	7.13×10^{-6}
	2			
6 Feb 89	1	4.57×10^{-6}		
	2			

The mean EF and associated 95 percent confidence interval are 9.94×10^{-6} , and 0 to 2.64×10^{-5} , respectively. The largest EF from the four estimates was 0.0000252 (2.52×10^{-5}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 25.2-millionths of a pound of phenol to be produced as a combustion product. For a one-ton (2,000 lb) TNT detonation, provided linear scaling applies, less than six one-hundredths of a pound ((0.0504 lb) or 22.9 g) of phenol would result. Phenol could not be detected by the SFC/MS method used by laboratory 2.

5.3.3.14.8 Dibenzofuran. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.24.

Table 5.24 Emission Factor Values for Dibenzofuran From the BangBox TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	1.02×10^{-7}		
	2	0.00	1.32×10^{-6}	5.62×10^{-7}
2 Feb 89	1		0.00	0.00
	2	6.49×10^{-9}	0.00	0.00
6 Feb 89	1	0.00		
	2	0.00	0.00	8.81×10^{-10}

The mean EF and associated 95 percent confidence interval are 1.53×10^{-7} and 0 to 3.85×10^{-7} , respectively. The largest EF from the 13 estimates was 0.00000132 (1.32×10^{-6}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 1.32-millionths of

a pound of dibenzofuran to be produced as a combustion product. For a one-ton (2,000 lb) TNT detonation, provided linear scaling applies, less than three one-thousandths of a pound ((0.00264 lb) or 1.20 g) of dibenzofuran would result.

5.3.3.14.9 Benz[a]anthracene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.25.

Table 5.25 Emission Factor Values for Benz[a]anthracene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	0.00		
	2	0.00	0.00	0.00
2 Feb 89	1		0.00	0.00
	2	0.00	0.00	0.00
6 Feb 89	1	0.00		
	2	0.00	0.00	0.00

All assays (when data from *complete* semi-VOST were used) showed no benz[a]anthracene. One quartz-fiber filter sample extracted by laboratory 2 and found to contain no benz[a]anthracene was later analyzed by laboratory 1, benz[a]anthracene was detected. The absence of benz[a]anthracene on all other samples analyzed provides strong evidence that this single detection may be an artifact.

5.3.3.14.10 Benzo[a]pyrene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.26.

Table 5.26 Emission Factor Values for Benzo[a]pyrene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	3.01×10^{-6}		
	2	0.00	0.00	0.00
2 Feb 89	1		1.37×10^{-6}	0.00
	2	0.00	0.00	0.00
6 Feb 89	1	2.70×10^{-7}		
	2	0.00	0.00	0.00

The mean EF and associated 95 percent confidence interval are 3.58×10^{-7} and 0 to 8.91×10^{-7} , respectively. The largest EF from the 13 estimates was 0.00000301 (3.01×10^{-6}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 3.01-millionths of a pound of benzo[a]pyrene to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than seven one-thousandths of a pound ((0.00602 lb) or 2.73 g) of benzo[a]pyrene would result. Benzo[a]pyrene was found on three of the four semi-VOST assays by laboratory 1; it was not found in the nine assays by laboratory 2.

5.3.3.14.11 Dibenz[a,h]anthracene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.27.

Table 5.27 Emission Factor Values for Dibenz[a,h]anthracene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	0.00		
	2	NA*	NA	NA
2 Feb 89	1		0.00	1.73×10^{-6}
	2	NA	NA	NA
6 Feb 89	1	0.00		
	2	NA	NA	NA

*Not applicable

The largest EF from the four estimates was 0.00000173 (1.73×10^{-6}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 1.73-millionths of a pound of Dibenz[a,h]anthracene to be produced as a combustion product. For a one-ton (2,000 lb) TNT detonation, provided linear scaling applies, less than four one-thousandths of a pound ((0.00346 lb) or 1.57 g) of Dibenz[a,h]anthracene would result. Dibenz[a,h]anthracene was not looked for by laboratory 2; it was found on one out of the four semi-VOST assays by laboratory 1.

5.3.3.14.12 N-Nitrosodiphenylamine. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.28.

Table 5.28 Emission Factor Values for N-Nitrosodiphenylamine From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	3.42×10^{-7}		
	2	0.00	0.00	8.03×10^{-8}
2 Feb 89	1		1.23×10^{-6}	8.80×10^{-7}
	2	0.00	0.00	0.00
6 Feb 89	1	0.00		
	2	0.00	0.00	0.00

The mean EF and associated 95 percent confidence interval are 1.95×10^{-7} and 0 to 4.36×10^{-7} , respectively. The largest EF from the 13 estimates was 0.00000123 (1.23×10^{-6}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 1.23-millionths of a pound of N-nitrosodiphenylamine to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than three one-thousandths of a pound ((0.00246 lb) or 1.12 g) of N-nitrosodiphenylamine would result.

5.3.3.14.13 Naphthalene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.29.

Table 5.29 Emission Factor Values for Naphthalene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	1.18×10^{-5}	1.50×10^{-4}	1.12×10^{-4}
2 Feb 89	1		NA	NA
	2	1.89×10^{-4}	0.00	0.00
6 Feb 89	1	NA		
	2	2.35×10^{-5}	0.00	0.00

^aNot applicable.

The mean EF and associated 95 percent confidence interval were 2.79×10^{-5} and 0 to 6.34×10^{-5} , respectively. The largest EF from the nine estimates was 0.000112 (1.50×10^{-4}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 150-millionths of a

pound of naphthalene to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than three tenths of a pound ((0.224 lb) or 102 g) of naphthalene would result. Naphthalene was not on the analyte list for laboratory 1; it was found by laboratory 2 on all direct semi-VOST assays, but only on one (the 31 January 1989) indirect semi-VOST assays.

5.3.3.14.14 1- & 2-Methylnaphthalene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.30.

Table 5.30 Emission Factor Values for 1- & 2-Methylnaphthalene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	1.67×10^{-6}	0.00	0.00
2 Feb 89	1		NA	NA
	2	0.00	0.00	0.00
6 Feb 89	1	NA		
	2	3.00×10^{-3}	0.00	0.00

^aNot applicable

The mean EF and associated 95 percent confidence interval are 3.52×10^{-6} , and 0 to 1.12×10^{-3} , respectively. The largest EF from the nine estimates was 0.00003 (3.00×10^{-3}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 30-millionths of a pound of 1- & 2-methylnaphthalene to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than seven one-hundredths of a pound ((0.0600 lb) or 27.2 g) of 1- & 2-methylnaphthalene would result. 1- & 2-Methylnaphthalene were not on the analyte list for laboratory 1. Laboratory 2 found it on 2 of 3 direct semi-VOST sampling trains but not on four indirect sampling trains.

5.3.3.14.15 1,3,5-Trinitrobenzene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.31.

Table 5.31 Emission Factor Values for 1,3,5-Trinitrobenzene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	0.00	2.75×10^{-9}	0.00
2 Feb 89	1		NA	NA
	2	0.00	0.00	0.00
6 Feb 89	1	NA		
	2	1.28×10^{-9}	0.00	0.00

^aNot applicable

The mean EF and associated 95 percent confidence interval are 4.47×10^{-10} and 0 to 1.19×10^{-9} , respectively. The largest EF from the nine estimates was 0.00000000275 (2.75×10^{-9}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 2.75-billionths of a pound of 1,3,5-trinitrobenzene to be produced as a combustion product, or released as a contaminant of the TNT. For one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than six-millionths of a pound ((0.0000540 lb) or 0.00245 g) of 1,3,5-trinitrobenzene would result. 1,3,5-trinitrobenzene was not on the list of analytes for assay by laboratory 1.

5.3.3.14.16 Biphenyl. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.32.

Table 5.32 Emission Factor Values for Biphenyl From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	0.00	0.00	0.00
2 Feb 89	1	NA	NA	
	2	0.00	0.00	0.00
6 Feb 89	1	NA		
	2	0.00	0.00	0.00

^aNot applicable

All assays from these three trials (when complete semi-VOST samples were analyzed) showed no biphenyl. In the eight TNT detonation trials on 8 February 1989, biphenyl was found on a quartz-

fiber filter used to accumulate all particles from all 8 detonations. The concentration reported was 15.1 ng/m³, well above the concentration (0.38 ng/m³) which would have been detectable (based on a lower detection limit of 1.15 ng/sample) on the 31 January 1989, and 2 and 6 February 1989 trials. Because there were no other biphenyl detections throughout the TNT testing program, the high concentration on this one filter is believed to be an artifact.

5.3.3.14.17 Phenanthrene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.33.

Table 5.33 Emission Factor Values for Phenanthrene From the BangBox TNT Detonation.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	1.85 x 10 ⁻⁷	0.00	0.00
2 Feb 89	1		NA	NA
	2	0.00	0.00	0.00
6 Feb 89	1	NA		
	2	0.00	0.00	0.00

^aNot applicable

The largest EF from the nine estimates was 0.000000185 (1.85 x 10⁻⁷). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 185-billionths of a pound of phenanthrene to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than four ten-thousandths of a pound ((0.000370 lb) or 0.169 g) of phenanthrene would result. Phenanthrene was not on the analyte list for laboratory 1; it was found on one of nine semi-VOST by laboratory 2.

5.3.3.14.18 2,5-Diphenyloxazole. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.34.

Table 5.34 EF Values for 2,5-Diphenyloxazole From the BB TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	1.42×10^{-5}	7.23×10^{-5}	4.75×10^{-10}
2 Feb 89	1		NA	NA
	2	2.95×10^{-8}	8.38×10^{-7}	3.45×10^{-8}
6 Feb 89	1	NA		
	2	8.56×10^{-6}	1.07×10^{-6}	1.26×10^{-7}

^aNot applicable

The mean EF and associated 95 percent confidence interval are 1.08×10^{-5} and 0 to 2.89×10^{-5} , respectively. The largest EF from the nine estimates was 0.0000723 (7.23×10^{-5}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 72-millionths of a pound of 2,5-diphenyloxazole to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than two tenths of a pound ((0.145 lb) or 65.6 g) of 2,5-diphenyloxazole would result. 2,5-Diphenyloxazole was not on the analyte list for laboratory 1.

5.3.3.14.19 1,1,3-Trimethyl-3-Phenylindane. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.35.

Table 5.35 EF Values for 1,1,3-Trimethyl-3-Phenylindane From the BB TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	1.60×10^{-7}	5.70×10^{-7}	4.90×10^{-7}
2 Feb 89	1		NA	NA
	2	7.73×10^{-8}	2.29×10^{-7}	4.64×10^{-8}
6 Feb 89	1	NA		
	2	6.39×10^{-9}	1.59×10^{-7}	2.40×10^{-7}

^aNot applicable

The mean EF and associated 95 percent confidence interval are 2.09×10^{-7} and 7.17×10^{-8} to 3.46×10^{-7} , respectively. The largest EF from the nine estimates was 0.00000057 (5.70×10^{-7}). Thus, for

every one-pound block of TNT detonated in the BB, one would expect no more than 570-billionths of a pound of 1,1,3-trimethyl-3-phenylindane to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than two one-thousandths of a pound ((0.00114 lb) or 0.517 g) of 1,1,3-trimethyl-3-phenylindane would result. 1,1,3-Trimethyl-3-phenylindane was not on the analyte list for laboratory 1.

5.3.3.14.20 Pyrene. Calculated EF values from data from the three trials (31 January 1989, 2 and 6 February 1989) are shown in Table 5.36.

Table 5.36 EF Values for Pyrene From the BB TNT Detonation Trials.

Date	Laboratory	Direct Sampling	Indirect Sampling	
			Nonhomogeneous	Homogeneous
31 Jan 89	1	NA ^a		
	2	2.02×10^{-7}	0.00	0.00
2 Feb 89	1	NA	NA	
	2	0.00	0.00	0.00
6 Feb 89	1	NA		
	2	8.27×10^{-8}	0.00	0.00

^aNot applicable

The mean EF and associated 95 percent confidence interval are 3.16×10^{-8} and 0 to 8.51×10^{-8} , respectively. The largest EF from the nine estimates was 0.000000202 (2.02×10^{-7}). Thus, for every one-pound block of TNT detonated in the BB, one would expect no more than 202-billionths of a pound of pyrene to be produced as a combustion product. For a one-ton (2,000-lb) TNT detonation, provided linear scaling applies, less than one five ten-thousandths of a pound ((0.000404 lb) or 0.183 g) of pyrene would result. Pyrene was not on the analyte list for laboratory 1.

5.3.4. Elements. Results from XRF analysis of filter samples obtained from direct sampling during the three TNT trials (31 January, 2 February, and 6 February 1989) were used in calculating the EFs presented in Table 5.37. A number of the target analytes were seen in these samples, but are not present in the TNT formulation. It is more likely that these analytes are from previous testing in the BB and from soil contaminants. The presence of these elements via the mechanism of particle resuspension from the walls and floor of the structure during passage of the detonation shock wave is not unexpected. There is no reason to expect that TNT was the source of any of the

target elements observed.

Table 5.37 EF for Target Elements in Direct Filter Samples from BB TNT Detonations.

Emission Factor					
Element	21 Jan 89	02 Feb 89	06 Feb 89	Mean	Std Dev
Cr	1.27×10^{-6}	3.52×10^{-6}	2.47×10^{-6}	2.42×10^{-6}	1.13×10^{-6}
Ni	2.54×10^{-6}	2.11×10^{-6}	2.12×10^{-6}	2.26×10^{-6}	2.47×10^{-7}
As	0.00	0.00	0.00	0.00	0.00
Pb	1.81×10^{-5}	1.97×10^{-5}	1.73×10^{-5}	1.84×10^{-5}	1.24×10^{-6}
Cd	2.86×10^{-6}	2.47×10^{-6}	2.82×10^{-6}	2.72×10^{-6}	2.18×10^{-7}
Sb	3.18×10^{-7}	7.05×10^{-7}	1.06×10^{-6}	6.94×10^{-7}	3.70×10^{-7}
Ba	8.27×10^{-4}	8.08×10^{-4}	9.31×10^{-4}	8.55×10^{-4}	6.62×10^{-5}

5.3.5. Ammonia. Solvent-filled bubblers were used during the BB trials to collect NH_3 . The mean calculated EF for NH_3 from the TNT trials of 31 January, 2 February, and 6 February 1989 was 1.1×10^{-4} . The values for NH_3 in the test samples were not statistically different from the readings in the background samples. The single high NH_3 reading (the principal contributor to the stated EF) was due to an unusually low background reading coinciding with a slightly high test sample reading. The detection level for ammonia corresponds to an emission factor of 0.00002.

5.3.6. Hydrogen Cyanide. Solvent-filled bubblers were aspirated during the BB trials so that HCN could be measured. No HCN was detected in samples from any of the BB trials. The detection level for hydrogen cyanide correspond to an emission factor of 0.00002.

5.4. Technical Assessment

5.4.1. The carbon mass balance method provided a stable (constant) EF over the duration of test, i.e. throughout the non-homogeneous and homogeneous mixed detonation cloud.

5.4.2. The cloud volume method did not give a dependable EF during the non-homogeneous mixed detonation cloud.

5.4.3. The variance associated with the EFs from the carbon mass balance method were equal to or smaller than the variances associated with the cloud volume method.

5.4.4. The sampling techniques used in the BB will provide valid measurements during subsequent OB/OD field tests with the FWAC.

5.4.5. The SFC/MS analysis method for the semivolatile (exotics) organics provided a means of detection of the thermally labile compounds.

5.4.6. The multiple detonation trial permitted detection of small quantities of the trace/exotic organic detonation products and also showed that the quartz-fiber filter collected most of the trace/exotic organic detonation products.

SECTION 6. DOUBLE-BASE PROPELLANT BURN

6.1. Objectives

6.1.1. To verify the validity of measurement and sampling techniques proposed for use on the FWAC (on subsequent OB/OD field tests).

6.1.2. To provide preliminary information (for planning purposes) on the morphology, composition, and size distribution of airborne particulate material and aerosols/vapors generated by propellant burns.

6.2. Test Procedures

6.2.1. Data Required

6.2.1.1. Photographic coverage from t-1 to t+35 min by a video camera inside the test chamber and by a video camera outside the chamber showing the BB as a whole.

6.2.1.2. HS video coverage from t-1 to t+35 min by a camera inside the test chamber.

6.2.1.3. Particle Size Distribution

6.2.1.3.1 One PMS probe (both ASASP 100X and FSSP 100X) particle size distribution reading each minute from t-45 to t+35 min.

6.2.1.3.2 One DMPS particle size distribution/5 min, from t-45 to t+35 min.

6.2.1.4. Analog data (5-s intervals) from t-45 to t+35 min from the following instruments:

6.2.1.4.1 Nephelometer.

6.2.1.4.2 CO₂ analyzer (± 2 ppm).

6.2.1.4.3 CO analyzer (± 0.1 ppm).

6.2.1.4.4 NO_x analyzer (± 5 ppb).

6.2.1.4.5 SO₂ analyzer (± 2 ppb).

6.2.1.4.6 O₃ analyzer (± 2 ppb).

6.2.1.4.7 PID for organic analyses (± 1 ppm).

6.2.1.5. Data Indicating Test Operating Conditions:

6.2.1.5.1 BB interior/exterior (ambient) differential pressure (± 6 mm of H₂O).

6.2.1.5.2 BB temperature ($\pm 0.5^\circ\text{C}$).

6.2.1.5.3 Bag sampler valve position.

6.2.1.5.4 Gas analyzer valve position.

6.2.1.6. Analog data (5-s intervals) from the following instruments on indirect (bag) samples taken at t-30, t+3, and t+15 min (or later, if need be, to ensure the homogeneity of the detonation products within the BB) for the period when the air bag was being pumped down:

6.2.1.6.1 CO₂ analyzer (± 2 ppm).

6.2.1.6.2 CO analyzer (± 0.1 ppm).

6.2.1.7. SFC/MS analysis of components (filters and resins) of the semi-VOST samplers operated inside the test chamber.

6.2.1.8. SFC/MS analysis of each component (filters and resins) of the semi-VOST samplers sampled from the 1.5-m³ air bag.

6.2.1.9. GC/MS analysis of components (filters and resins) of the semi-VOST samplers operated inside the test chamber.

6.2.1.10. GC/MS analysis of components (filters and resins) of the semi-VOST samples collected from the 1.5-m³ air bag.

6.2.1.11. XRF elemental analysis of Teflon™ filters exposed inside the BB from t-45 to t-15 min and from t+2 to t+35 min.

6.2.1.12. XRF elemental analysis of Teflon™ filter samples exposed to the air drawn from the bag samples obtained at t-30, t+3, t+15, and t+30 min.

6.2.1.13. SEM analysis of particles from Nuclepore™ filters exposed within the test chamber from t-45 to t-15 min and from t+2 to t+35 min.

6.2.1.14. SEM analysis of Nuclepore™ filters exposed to the air drawn from the bag samples obtained at t-30, t+3, and t+15 min.

6.2.1.15. Analyses for THC, CH₄, C₂-C₁₀, C₆H₆, CO, CO₂ and H₂ drawn from inside the chamber at t-30, t+3, t+15, and t+30 and contained in 6-L sampling canisters.

6.2.1.16. Analyses for HCN and NH₃ in bubbler samples obtained from t-45 to t-15 min and from t+2 to t+35 min.

6.2.1.17. semi-VOST flow rate as measured before and after the burn trial.

6.2.1.18. Differential pressure measured at 5-min intervals from t+5 to t+30 min.

6.2.1.19. Exterior background air samples (in 6-L canisters) taken at t-5, t+3, and t+15 min.

6.2.2. Data Acquisition Procedures

6.2.2.1. After being weighed, 454 g of NOSIH-AA2 double-base propellant end mill chips and 26.6 g of ethyl cellulose inhibitor (propellant wrapping material) were placed in a stainless steel bowl 103 cm diameter by 12.7 cm deep. After one Atlas™ electric match was inserted into the mix, the bowl was covered by a coarse steel mesh to prevent thermal updrafts from lofting burning segments of propellant out of the bowl. The propellant, a standardized item manufactured by the Radford Army Ammunition Plant, Radford, Virginia, is composed primarily of nitrocellulose and nitroglycerin (Table 6.1). Samples of propellant and inhibitor, were taken for subsequent laboratory analysis.

Table 6.1 Nominal Composition of NOSIH-AA2 Double-Base Propellant with Ethyl Cellulose Wrapping Material.

Component	Weight		Carbon in Component	
	Percent	Grams	Percent	Grams
Nitrocellulose	49.2	231.55	27.04	62.61
Nitroglycerin	37.2	175.24	15.87	27.81
Triacetin	2.6	12.26	49.54	6.07
Di-n-propyl adipate	1.9	9.08	67.28	6.11
2-Nitrodiphenylamine	1.5	7.26	62.58	4.54
Lead λ -salicylate	1.5	6.81	34.92	2.38
Lead resorcyate	0.5	2.27	32.76	0.74
Copper salicylate	1.9	9.08	49.78	4.52
Candelilla wax	<0.1	0.45	85.23	0.38
Ethyl cellulose	3.6	17.1	58.51	10.01
Total		471.1		125.17

6.2.2.2. An electric match, identical to the match used to ignite the propellant was placed in a sample jar for subsequent laboratory assay.

6.2.2.3. Flow-rate readings were taken of semi-VOSTs, HCN and NH₃ bubblers, and XRF and SEM filters.

6.2.2.4. Real-time samplers/analyzers operated throughout the subtest. Sampling results were collected on data loggers and reduced to engineering units by SNL.

6.2.2.5. One mixing fan blade was swabbed and rinsed with solvent before the trial began. Both swab material (paper towel) and solvent were collected and sealed in sample jars for subsequent laboratory assay. The cleaned fan blade was identified, by scratched marking, to permit locating the cleaned and sampled area following the burn. Post-burn sampling used identical procedures, except fan-blade identification marking was not done.

6.2.2.6. Mixing fans were turned on prior to ignition of the propellant, and operated continuously throughout the subtest in order to reduce the possibility of overheating or burning the BB fabric.

6.2.2.7. The pressure differential between the chamber and external ambient atmosphere was constantly monitored and the blower damper adjusted to achieve as constant a pressure differential as possible.

6.2.3. Analytical Procedure

6.2.3.1. Sample Distribution.

6.2.3.1.1 Filters and cartridges were individually sealed in aluminum foil, labeled so as to permit positive identification, and prepared for shipment to designated assay laboratories. To ensure prompt and undamaged delivery, all samples were hand-carried by courier.

6.2.3.1.2 All 6-L and 0.85-L canisters were packaged in shock-resistant containers and shipped, via common carrier, to OGC for assay or archiving.

6.2.3.1.3 All other samples, which included swabs, propellant, propellant inhibitor, and electric match, were packaged in sealed containers and delivered to AWL for assay.

6.2.3.2. Detection and Identification

6.2.3.2.1 Organics. The GC/MS was used to identify and quantitate real-time gas and volatile organic species. Both SFC/MS and GC/MS were used to detect and quantitate other organic species. SFC/MS and GC/MS procedures used in this analysis are outlined in Volume 3.

6.2.3.2.2 Elementals. XRF was used to determine elemental content. Procedures are outlined in Volume 3.

6.2.3.2.3 Carbon. Pyrolysis/combustion techniques, were used for carbon analysis, (Volume 3).

6.2.3.2.4 Total Suspended Particles (TSP). TSP were determined by gravimetric analysis.

6.3. Test Findings

6.3.1. Carbon Mass Balance.

The available double-base propellant carbon mass was estimated by two methods: (1) the amount released (calculation based on the double-base propellant molecular formula; and from elemental analysis of propellant samples by M-H-W Laboratories, Phoenix, Arizona) and (2) the amount measured (accounting for all carbon-containing products through analysis of aerosol and particulate samples taken during the trial). The mass of double-base propellant burned was 454 g; the mass of ethyl cellulose burned was 17.1 g (9.5 g of ethyl cellulose did not burn).

6.3.1.1. The percent of available carbon calculated from information on the molecular formula of this double-base propellant and the molecular formula of ethyl cellulose was 26.57 percent (Table 6.1).

6.3.1.2. The M-H-W Laboratories' analysis of one double-base propellant sample and two ethyl cellulose samples gave the following percentages for the elemental carbon:

Double-base propellant sample:	25.39 percent carbon
Ethyl cellulose sample #1:	60.70 percent carbon
Ethyl cellulose sample #2:	60.51 percent carbon

6.3.1.2.1 A weighted average (based on the actual amounts of propellant and ethyl cellulose in the burn mix) for the double-base burn material yields 26.67 percent carbon, a value very close to that estimated using the molecular formula.

6.3.1.3. Analysis of the air samples taken during the trial provided an estimate of the total carbon mass based on summation of the carbon contained in CO_2 , CO, the organic carbon, and the elemental carbon. The carbon mass contributed by CO_2 was estimated from the results of real-time (continuously monitored) instrument sampling. CO_2 concentration estimates immediately after detonation were derived by extrapolation of the fitted exponential curve (fit to the concentration data from the homogeneous period of sampling) to detonation time ($t = 0$). The carbon contributed from CO was estimated from the results of the 6-L canister samples with extrapolation of the fitted exponential curve of concentration values to detonation time zero. The organic carbon and elemental carbon contributions were estimated by thermal analysis of a 1-cm² sample taken from the quartz-fiber filter of the semi-VOST. The analysis incorporated a two-step volatilization and combustion process to differentiation between volatile and elemental carbon on the filter.

6.3.1.3.1 The total carbon mass derived from CO_2 , CO, organic carbon, and elemental carbon as a result of the double-base burn conducted 9 February 1989 was 132.13 g. This carbon mass value is 6 percent greater than that calculated from the propellant formulation (125.17 g) and is 5 percent greater than the amount calculated based on the carbon analysis done by M-H-W Laboratories (125.63 g). For purposes of calculating EF values, 125.17 g, based on data for the formulation of the double-base propellant, was used.

6.3.2. Emission Factors.

EFs of compounds and elements detected from the double-base burn were calculated by two methods; the carbon balance method, (described in paragraph 5.3.2.1) and the cloud volume method, is described in paragraph 5.3.2.3.

6.3.2.1. Real Time Continuously Monitored Gases Analyzed by SNL. Continuously monitored gases CO_2 , NO, NO_2 , and SO_2 by real-time sampling. The concentration data used in computing EFs were those from the homogeneous sampling period (after 3 min of fanning). The resulting EFs, calculated by both methods, for these continuously monitored gases are given in Table 6.2.

Table 6.2 EF for the Continuously Monitored Gases CO₂, NO, NO₂, and SO₂ From the Double-Base Propellant Burn, Calculated by the Carbon Balance and by the C-V Method.

Method	Sample Source	Compound			
		CO ₂	NO	NO ₂	SO ₂
Carbon Balance	Direct	9.70 x 10 ⁻¹	2.34 x 10 ⁻²	2.02 x 10 ⁻⁴	3.22 x 10 ⁻⁴
	Indirect	9.70 x 10 ⁻¹	1.90 x 10 ⁻²	0.00	3.21 x 10 ⁻⁴
		9.70 x 10 ⁻¹	1.90 x 10 ⁻²	2.42 x 10 ⁻³	3.10 x 10 ⁻⁴
	Average	9.70 x 10 ⁻¹	1.41 x 10 ⁻²	8.75 x 10 ⁻⁴	2.11 x 10 ⁻⁴
	Std Dev	0.00	2.54 x 10 ⁻³	1.34 x 10 ⁻³	6.66 x 10 ⁻⁶
Cloud Volume	Direct	1.02	2.47 x 10 ⁻²	2.13 x 10 ⁻⁴	3.40 x 10 ⁻⁴
	Indirect	9.49 x 10 ⁻¹	1.86 x 10 ⁻²	0.00	3.14 x 10 ⁻⁴
		1.13	2.21 x 10 ⁻²	2.81 x 10 ⁻³	3.60 x 10 ⁻⁴
	Average	1.03	2.18 x 10 ⁻²	1.01 x 10 ⁻³	3.38 x 10 ⁻⁴
	Std Dev	9.12 x 10 ⁻²	3.06 x 10 ⁻³	1.56 x 10 ⁻³	2.31 x 10 ⁻³

6.3.2.1.1 A comparison of EFs calculated by the carbon balance method with those calculated by the cloud volume method showed no statistically significant differences for these four gases.

6.3.2.2. Volatile Compounds. Samples for CO₂, CO, volatile compounds (methane, acetylene, and benzene and a series of paraffins, olefins, nonbenzene aromatics, and terpenes) were collected by 6-L canisters during the homogeneous period. The list of compounds considered within these groupings are outlined in Table 5.7. The EFs for these groups of compounds are listed in Table 6.3.

Table 6.3 EF for the Canister-Sampled CO₂, CO, and Volatile Organics from the Double-Based Propellant Burn, Calculated by the Carbon Balance Method and by the Cloud Volume Method.

Method	Sample Source	Volatile Organics (Exotics)							
		Carbon Dioxide	Carbon Monoxide	Methane	Paraffins	Olefins	Non-Benzene Aromatics	Acetylene	Renzene
Carbon Balance	Direct	9.70 x 10 ⁻¹	9.12 x 10 ⁻⁴	5.92 x 10 ⁻³	9.77 x 10 ⁻⁶	6.75 x 10 ⁻³	5.72 x 10 ⁻⁶	1.81 x 10 ⁻³	1.12 x 10 ⁻³
		9.68 x 10 ⁻¹	9.54 x 10 ⁻⁴	6.68 x 10 ⁻³	1.29 x 10 ⁻³	6.86 x 10 ⁻³	1.20 x 10 ⁻³	1.93 x 10 ⁻³	1.16 x 10 ⁻³
		9.70 x 10 ⁻¹	9.12 x 10 ⁻⁴	6.87 x 10 ⁻³	1.24 x 10 ⁻³	7.62 x 10 ⁻³	1.56 x 10 ⁻³	2.06 x 10 ⁻³	1.27 x 10 ⁻³
		9.70 x 10 ⁻¹	8.70 x 10 ⁻⁴	7.26 x 10 ⁻³	5.19 x 10 ⁻³	7.94 x 10 ⁻³	9.85 x 10 ⁻³	2.18 x 10 ⁻³	1.03 x 10 ⁻³
	Average	9.69 x 10 ⁻¹	9.12 x 10 ⁻⁴	6.68 x 10 ⁻³	2.18 x 10 ⁻³	7.29 x 10 ⁻³	3.30 x 10 ⁻³	1.99 x 10 ⁻³	1.15 x 10 ⁻³
	Indirect	9.27 x 10 ⁻¹	7.97 x 10 ⁻⁴	5.58 x 10 ⁻³	6.98 x 10 ⁻³	5.96 x 10 ⁻³	0.00	1.68 x 10 ⁻³	4.78 x 10 ⁻⁶
		7.82 x 10 ⁻¹	6.30 x 10 ⁻⁴	4.53 x 10 ⁻³	8.80 x 10 ⁻⁶	6.00 x 10 ⁻³	6.29 x 10 ⁻⁷	1.76 x 10 ⁻³	1.01 x 10 ⁻³
Cloud Volume	Average	8.54 x 10 ⁻¹	7.13 x 10 ⁻⁴	5.06 x 10 ⁻³	3.93 x 10 ⁻³	5.98 x 10 ⁻³	3.14 x 10 ⁻⁷	1.72 x 10 ⁻³	7.42 x 10 ⁻⁶
	Direct	9.66 x 10 ⁻¹	9.09 x 10 ⁻⁴	5.90 x 10 ⁻³	9.74 x 10 ⁻⁶	6.72 x 10 ⁻³	5.70 x 10 ⁻⁶	1.80 x 10 ⁻³	1.12 x 10 ⁻³
		9.20 x 10 ⁻¹	9.07 x 10 ⁻⁴	6.35 x 10 ⁻³	1.23 x 10 ⁻³	6.52 x 10 ⁻³	1.14 x 10 ⁻³	1.83 x 10 ⁻³	1.10 x 10 ⁻³
		8.99 x 10 ⁻¹	8.46 x 10 ⁻⁴	6.37 x 10 ⁻³	1.15 x 10 ⁻³	7.07 x 10 ⁻³	1.44 x 10 ⁻³	1.91 x 10 ⁻³	1.18 x 10 ⁻³
		9.22 x 10 ⁻¹	8.27 x 10 ⁻⁴	6.91 x 10 ⁻³	4.94 x 10 ⁻³	7.55 x 10 ⁻³	9.37 x 10 ⁻³	2.07 x 10 ⁻³	9.81 x 10 ⁻⁶
	Average	9.27 x 10 ⁻¹	8.72 x 10 ⁻⁴	6.38 x 10 ⁻³	2.07 x 10 ⁻³	6.97 x 10 ⁻³	3.13 x 10 ⁻³	1.90 x 10 ⁻³	1.09 x 10 ⁻³
	Indirect	9.07 x 10 ⁻¹	7.80 x 10 ⁻⁴	5.47 x 10 ⁻³	6.83 x 10 ⁻³	5.84 x 10 ⁻³	0.00	1.65 x 10 ⁻³	4.68 x 10 ⁻⁶
		9.08 x 10 ⁻¹	7.32 x 10 ⁻⁴	5.27 x 10 ⁻³	1.02 x 10 ⁻³	6.97 x 10 ⁻³	7.30 x 10 ⁻⁷	2.04 x 10 ⁻³	1.17 x 10 ⁻³
	Average	9.08 x 10 ⁻¹	7.56 x 10 ⁻⁴	5.37 x 10 ⁻³	3.93 x 10 ⁻³	6.41 x 10 ⁻³	3.65 x 10 ⁻⁷	1.85 x 10 ⁻³	8.18 x 10 ⁻⁶

6.3.2.2.1 A comparison of the mean EF values calculated for 6-L canister-sampled CO₂ from the chamber (direct sampling) showed a statistical difference between the carbon balance method (EF = 0.969) and the cloud volume method (EF = 0.927); however, no differences were found between methods for the EFs calculated for any of the other compounds or groups of compounds.

6.3.2.2.2 The range of EFs noted for the volatile hydrocarbons are shown in Table 6.4.

Table 6.4 EF for the Volatile Hydrocarbons.

Compound	Direct	Indirect
Methane	5.90×10^{-5} to 7.26×10^{-5}	4.53×10^{-5} to 5.58×10^{-5}
Paraffins	9.74×10^{-6} to 5.19×10^{-5}	8.80×10^{-6} to 6.98×10^{-5}
Acetylene	1.80×10^{-5} to 2.18×10^{-5}	1.65×10^{-5} to 2.04×10^{-5}
Olefins	6.52×10^{-5} to 7.94×10^{-5}	5.84×10^{-5} to 6.97×10^{-5}
Benzene	9.81×10^{-6} to 1.27×10^{-5}	4.68×10^{-6} to 1.01×10^{-5}
Nonbenzene aromatics	5.72×10^{-6} to 9.85×10^{-5}	0 to 7.30×10^{-7}

6.3.2.3. No EFs are shown for the terpene groups of compounds because they were not detected in the double-base propellant burn trial samples. Using the largest calculated EF's, the maximum quantities of emissions from each category of volatile organics expected to be produced or released from a double-base propellant burn were calculated and are shown in Table 6.5.

Table 6.5 Maximum Quantities of Emissions from each Category of Volatile Organics expected to be Produced or Released from a Double-Base Propellant Burn

Compound	Source Strength (1-lb burn) (lb)	Source Strength (2000-lb burn) (lb/g)
Methane	7.26×10^{-5}	0.145/65.9
Paraffins	6.98×10^{-5}	0.140/63.3
Acetylene	2.18×10^{-5}	0.0436/19.8
Olefins	7.94×10^{-5}	0.159/72.0
Benzene	1.27×10^{-5}	0.0254/11.5
Nonbenzene aromatics	9.85×10^{-5}	0.197/89.4
Totals	3.55×10^{-4}	0.71/320

6.3.2.4. Semivolatile Organics.

6.3.2.4.1 The test design provided data for calculation of EFs for semivolatile organics from the direct semi-VOST, the indirect-inhomogeneous semi-VOST, and the indirect-homogeneous semi-semi-VOST. The net amounts of semivolatile organic compounds detected, calculated by summing the amounts found in each of the three components of the sampling train after correcting for background, were converted to EFs for each of the following compounds:

2,6-Dinitrotoluene
4-Nitrophenol
2-Nitronaphthalene
2,4,6-Trinitrotoluene
Phenol
Dibenzofuran
Benzo[a]pyrene
N-Nitrosodiphenylamine

6.3.2.4.2 The following compounds, although looked for, were never found:

2,4-Dinitrotoluene
1-Nitropyrene
1,6-Dinitropyrene
2-Naphthalamine
Diphenylamine
Benz[c]acridine
Benz[a]anthracene
Dibenz[a,h]anthracene
Naphthalene
4-Nitrosodiphenylamine
2-Nitrodiphenylamine
4-Nitrodiphenylamine
Nitroglycerin

Resorcinol
Di-n-propyl adipate
Triacetin
Salicylic acid

6.3.2.4.3 The 25 target analyte organic compounds cited in the two lists above are those which were searched for and, when found, measured by GC/MS and/or SFC/MS selected-ion monitoring techniques. Basically, the target analyte list consisted of: some of the components of the double-base propellant mixture which may still be present after the burn; and combustion product compounds which, if present in high concentration, would be of environmental concern.

6.3.2.4.4 The analysis results for each of the eight compounds that were detected at one time or another during the analyses performed are discussed separately in subparagraphs below. Sampling provided data for two EF estimates for each compound. One of the EF estimates was from laboratory 1 indirect-homogeneous sampling and the second was from laboratory 2 direct sampling.

a. 2,6-Dinitrotoluene. The two estimates of EFs are 1.43×10^{-8} , for the samples derived from indirect-homogeneous sampling and zero from samples derived from direct sampling. Using the 1.43×10^{-8} value and assuming a one-pound double-base propellant burn, no more than 14.3-billionths of a pound of 2,6-dinitrotoluene is expected to be produced as a combustion product. For a one-ton (2,000-lb) double-base propellant burn, provided linear scaling applies, less than one ten-thousandths of a pound ((0.0000286 lb) or 0.0130 g) of 2,6-dinitrotoluene would result. Because 8 TNT detonations were conducted the day before this propellant burn, and 2,6-dinitrotoluene was noted in both background and test samples, this small quantity may have resulted from contamination from the prior-day TNT trial.

b. 4-Nitrophenol. The two estimates of EFs are 6.87×10^{-7} from samples derived from the indirect-homogeneous sampling samples and 8.61×10^{-9} for the direct sampling samples. Using the 6.87×10^{-7} value and assuming a one-pound double-base propellant burn, no more than 687-billionths of a pound of 4-nitrophenol is expected to be produced as a combustion product. For a one-ton (2,000-lb) double-base propellant burn, provided linear scaling applies, less than two one-thousandths of a pound ((0.00137 lb) or 0.622 g) of 4-nitrophenol would result. As noted in

the TNT trial results, 4-nitrophenol was detected at these extremely low levels in most background, and test samples, and in travel blanks. Thus, it is probable that most of the 4-nitrophenol detected was not due to propellant combustion.

c. 2-Nitronaphthalene. The two estimates of EFs are 0 for the indirect-homogeneous sampling samples and 5.43×10^{-8} for the direct sampling samples. Using the 5.43×10^{-8} value and assuming a one-pound double-base propellant burn, no more than 54.3-billionths of a pound of 2-nitronaphthalene is expected to be produced as a combustion product. For a one-ton (2,000-lb) double-base propellant burn, provided linear scaling applies, less than two ten-thousandths of a pound ((0.000109 lb) or 0.0493 g) of 2-nitronaphthalene would result. Because this compound is found as a combustion product from TNT detonation in concentrations substantially higher than noted here (mean EF = 1.64×10^{-7}) and because eight detonations were conducted the day prior to this propellant burn, it is possible that most of this small amount of 2-nitronaphthalene resulted from residual contamination from the 8 February 1989 test.

d. 2,4,6-Trinitrotoluene. The two estimates of EF are 5.01×10^{-8} for the indirect-homogeneous sampling samples and 0 for the direct sampling samples. Using the 5.01×10^{-8} value and assuming a one-pound double-base propellant burn, no more than 50.1-billionths of a pound of 2,4,6-trinitrotoluene is expected to be produced as a combustion product or released as a contaminant. Thus for a one-ton(2,000 lb) double-base propellant burn, provided linear scaling applies, less than two one-thousandths of a pound ((0.000100 lb) or 0.0454 g) of 2,4,6-trinitrotoluene may result. For the same reasons as noted in the above discussions of 2,6-dinitrotoluene and 2-nitronaphthalene, the small quantity of TNT detected was probably residual contamination from prior-day TNT testing.

e. Phenol. The one estimate of EF was 4.39×10^{-6} from analysis of the indirect-homogeneous sampling data. Using this value of 4.39×10^{-6} and assuming a one-pound double-base propellant burn, no more than 4.39-millionths of a pound of phenol is expected to be produced as a combustion product or released as a contaminant. For a one-ton (2,000-lb) double-base propellant burn, provided linear scaling applies, less than nine one-thousandths of a pound ((0.00878 lb) or 3.98 g) of phenol would result. Because phenol is a widespread air contaminant and was found often in the background samples in concentrations similar to those found in actual test samples, it

is uncertain that more than a small fraction of the detected phenol resulted from the burn itself.

f. Dibenzofuran. The two estimates of EFs are 2.86×10^{-7} from analysis of the indirect-homogeneous sampling data and 2.25×10^{-7} from the direct sampling data. Using the 2.86×10^{-7} value and assuming a one-pound double-base propellant burn, no more than 286-billionths of a pound of dibenzofuran is expected to be produced as a combustion product of the double-base propellant burn. For a one-ton (2,000-lb) double-base propellant burn, provided linear scaling applies, less than six ten-thousandths of a pound ((0.000572 lb) or 0.259 g) of dibenzofuran would result.

g. Benzo[a]pyrene. The one estimate of EF was 8.96×10^{-7} from analysis of the indirect-homogeneous sampling data. Using this value, and assuming a one-pound double-base propellant burn, no more than 896-billionths of a pound of benzo[a]pyrene is expected to be produced as a combustion product or released as contaminant. For a one-ton (2,000-lb) double-base propellant burn, provided linear scaling applies, less than two one-thousandths of a pound ((0.00179 lb) or 0.813 g) of benzo[a]pyrene would result.

h. N-Nitrosodiphenylamine. The two estimates of EF, are 0 for the indirect-homogeneous sampling and 1.45×10^{-6} for the direct sampling. Using the 1.45×10^{-6} value and assuming a one-pound double-base propellant burn, no more than 1.45-millionths of a pound of N-nitrosodiphenylamine is expected to be produced as a combustion product. For a one-ton (2,000-lb) double-base propellant burn, provided linear scaling applies, less than three one-thousandths of a pound ((0.0029 lb) or 1.32 g) of N-nitrosodiphenylamine would result.

6.3.2.5. Elements. Results from XRF analysis of filter samples obtained from direct sampling taken during the double-base propellant burn of 9 February 1989 were used to calculate the EFs presented in Table 6.6. Only copper and lead were observed, and both of these metals are present in the propellant formulation. The observed EF for copper, 3.71×10^{-3} , corresponds to 7.42 lbs (3.36 kg) per ton. The copper content of this propellant is 7.15 lbs per ton; thus, the recovery of copper was 104 percent. (Copper is not on the target analyte list.) The EF for lead is 1.27×10^{-2} , or 25.4 lbs (11.5 kg) per ton of double-base propellant. Since the lead content of the propellant is 18.9 lbs per ton, the recovery was 134 percent, indicating that the EF is high because excess lead

was found on the filter sample.

Table 6.6 EF for Target Elements and Copper in Direct Filter Samples from Double-Base Propellant Burn in the BB.

Element	Emission Factor
Chromium	0.00 ^a
Nickel	0.00
Copper	3.71×10^{-3}
Arsenic	0.00
Lead	1.27×10^{-2}
Cadmium	0.00
Tin	0.00
Barium	0.00

^aAn EF of zero means the concentration of an element if present was below the detection level.

6.3.2.6. Ammonia. Solvent-filled bubblers aspirated during the double-base propellant burn trial did not yield a detectable quantity of NH_3 , so the emission factor is zero.

6.3.2.7. Hydrogen Cyanide. Solvent-filled bubblers were used during the BB trials to collect HCN. No HCN was detected in the sample from the double-base propellant burn trial.

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SECTION 7. COMPOSITE PROPELLANT BURN

7.1. Objectives

7.1.1. To verify the validity of measurement and sampling techniques proposed for use on the FWAC during subsequent OB/OD field tests.

7.1.2. To provide preliminary information (for planning purposes) on the morphology, composition, and size distribution of airborne particulate material generated by detonations and propellant burns.

7.2. Test Procedure

7.2.1. Data Required

7.2.1.1. Video coverage, including HS video, from t-1 to t+35 min by a camera inside the test chamber.

7.2.1.2. Particle Size Distribution.

7.2.1.2.1 One PMS (both ASASP 100X and FSSP 100X) particle size distribution reading each min from t-45 to t+35 min.

7.2.1.2.2 One DMPS particle size distribution reading every 5 min, from t-45 to t+35 min.

7.2.1.3. Analog data (5-s intervals), from t-45 to t+35 min, from the following instruments:

7.2.1.3.1 Nephelometer.

7.2.1.3.2 CO₂ analyzer (± 2 ppm).

7.2.1.3.3 CO analyzer (± 0.1 ppm).

7.2.1.3.4 NO_x analyzer (± 5 ppb).

7.2.1.3.5 SO₂ analyzer (± 2 ppb).

7.2.1.3.6 O₃ analyzer (± 2 ppb).

7.2.1.3.7 PID for organic analyses (± 1 ppm).

7.2.1.4. Data Indicating Test Operating Conditions:

7.2.1.4.1 BB interior/exterior (ambient) differential pressure (± 6 mm of H₂O).

7.2.1.4.2 BB temperature ($\pm 0.5^\circ\text{C}$).

7.2.1.4.3 Gas analyzer valve position.

7.2.1.5. GC/MS and/or GC/MS analysis of each component (filters and resins) of the semi-VOST samplers operated inside the test chamber for collection of volatile, semivolatile, and nonvolatile organics.

7.2.1.6. XRF elemental analysis of Teflon™ filters exposed inside the BB from t-45 to t-15 min and from t+2 to t+35 min.

7.2.1.7. XRF elemental analysis of Teflon™ filter samples exposed to the air drawn from the bag samples obtained at t-30, t+3, t+15, and t+30 min.

7.2.1.8. SEM of particles from Nuclepore™ filters exposed within the test chamber from t-45 to t-15 min and from t+2 to t+35 min.

7.2.1.9. Analyses for HCN, NH₃, and HCl in bubbler samples obtained from t-45 to t-15 min and from t+2 to t+35 min.

7.2.1.10. Determination of HCl (Cl) levels following a pretest using an Interscan™ HCl Series 4000 meter and MSA™ chlorine detector tubes.

7.2.1.11. Dibenzodioxin and Dibenzofuran analyses using Pallflex 2500™ quartz tissue filters in series with polyurethane foam (PUF) filters.

7.2.1.12. Semi-VOST flow rates as measured before and after the burn trial.

7.2.2. Data Acquisition Procedures

7.2.2.1. On the day preceding the composite propellant burn subtest, a pretest was conducted to provide the basis for determining respiratory safety measures necessary to protect test personnel entering the BB after the subtest. To this end, 115 g (0.25 lb) of composite propellant was burned in the BB.

7.2.2.2. Early the next morning, BCD personnel collected 2-h background air samples inside the BB using two samplers operating simultaneously. Each sampler had one quartz tissue filter in series with a PUF filter, a setup paralleling that used in the semi-VOST.

7.2.2.3. During final preparations for the full-scale burn, test personnel placed 448.41 g of Mk 6 Product Improvement Program (PIP) Mix 88-P-217 propellant (Table 7.1) in a SS bowl 103-cm diameter by 12.7-cm depth (16-in diameter by 5-in depth). Two Atlas™ electric matches were inserted into the mix, and the bowl was covered by a coarse steel mesh in a fashion similar to that used in the double-base propellant subtest. The PIP mix, an experimental propellant under development by the Naval Ordnance Station, Indian Head, Maryland,(NOSIH) was selected for this subtest because of its high chlorine content.

Table 7.1 Nominal Composition of Propellant Mix 88-P-217, Mk 6 Product Improvement Program.

Component	Weight		Carbon in Component	
	Percent	Grams	Percent	Grams
Hydroxyl-terminated polybutadiene	8.0	35.88	88.82	31.87
2,2-methylene bis (4-methyl)-6-t-butyl phenol	0.2	0.89	81.13	0.72
Dioctyl sebacate	4.5	20.18	73.19	14.77
Phenyl di-isodecyl phosphite	0.2	0.89	71.20	0.63
5-ethyl-1,3-diglycidyl-5-methyl hydantoin diepoxide	0.3	1.35	56.68	0.77
Aluminum oxide	1.0	4.48	0.00	0.00
Carbon	0.1	0.45	100.00	0.45
Ferric acetylacetonate	<0.1	0.02	51.01	0.01
Ammonium perchlorate	85.0	381.15	0.00	0.00
Diethylenetriamine	0.1	0.36	46.56	0.17
Isophorone di-isocyanide	0.6	2.76	64.84	1.79
TOTAL	448.41		51.18	

7.2.2.4. Flow-rate readings were taken of semi-VOSTs, bubblers, and Teflon™ and Nuclepore™ filters before and after sampling.

7.2.2.5. Two bubblers, in series, were used to sample for HCN, NH₃, and HCl. Each of these bubbler series was dedicated to sampling for one of the target compounds. The HCl and HCN bubblers used an aqueous solution of 10 percent sodium hydroxide. The NH₃ bubbler used an aqueous solution of 10 percent normal sulfuric acid. The bubblers were manually turned on; turnoff was controlled by an automatic timer. Background sampling was conducted for 30 min, from t-56.5 to t-26.5 min. Sampling with fresh bubblers was conducted for 34 min starting at t-3 min.

7.2.2.6. Real-time samplers/analyzers, except the DMPS (which was not used), operated continuously throughout the subtest. Sampling results were collected on data loggers and reduced to engineering units by SNL.

7.2.2.7. Mixing fans, turned on before ignition, were operated until homogeneity was achieved and there was no further possibility of overheating or burning the BB fabric. The fan blade that was

marked for sampling in prior subtests was cleaned by swabbing and rinsing with methylene glycol before the 441.41 g propellant sample was ignited. Both swab and solvent were collected and sealed in sample jars for subsequent laboratory assay. The fan blade was sampled, again using identical procedures, after the composite propellant had been burned.

7.2.2.8. The 1.5-m³ Velostat™ bag system was used to collect the aerosol through a 10-cm diameter aluminum sampling probe that extended into the chamber. The bag was filled and the collected aerosol pumped through two semi-VOST, one Teflon™ filter, one Nuclepore™ filter, and real-time monitors after homogeneity was believed to have been achieved. Procedures for operating this system are found in Volume 3.

7.2.2.9. The semi-VOSTs collected background samples directly from the chamber for approximately 34 min, from t-57.2 to t-23.5 min. Effective direct emission sampling was conducted for 38 min starting at t-3 min. Indirect chamber sampling through the Velostat™ bag, was conducted at t-46 and t+6 min.

7.2.2.10. Teflon™ filters were used to collect samples directly from the BB for XRF analysis, and Nuclepore™ filters similarly collected samples for SEM analysis. Background samples were collected for approximately 34 min, from t-57.2 to t-23.5 min. Effective direct emission sampling was conducted for approximately 38 min starting at t=0. Indirect chamber sampling by Teflon™ and Nuclepore™ filters, through the Velostat™ bag, was conducted at t-21, t+9, t+19 and t+39 min.

7.2.2.11. Direct chamber air sampling by 6-L canister was conducted at t-45, t+2, t+8, t+16, and t+35 min.

7.2.2.12. Indirect chamber air sampling by 6-L canister, using the Velostat™ bag, was conducted at t+2, t+6, t+17, and t+36 min.

7.2.2.13. Exterior background air samples were collected by 6-L canister at t-46 and t+5 min.

7.2.2.14. SF₆ sampling by 0.85-L canister, from locations 2 m above the floor and at the top of the chamber, was conducted at 2-min intervals from t+2 to t+30 min.

7.2.2.15. Effective dioxin sampling was conducted for 2 h starting at $t=0$.

7.2.2.16. A metal clip (identical in specification to clips used during the double-base propellant burn subtest) used to anchor igniter electrical wire leads was taken as a sample for laboratory assay.

7.2.2.17. The HS video camera, equipped with a 5.7-mm lens and 1/250-s shutter, took video pictures of the burn at the speed of 5000 frames/s.

7.2.2.18. The pressure differential between the chamber interior and the external ambient atmosphere was constantly monitored and adjusted to ensure adequate flow rates into the Velostat™ bag. When the differential dropped below 18 mm of H₂O, the inflation blower damper was adjusted to return the pressure differential to desired levels.

7.2.2.19. The chamber was not entered after the burn trial until dioxin sampling had concluded ($t+120$ min).

7.2.3. Analytical Procedures

7.2.3.1. Sample Distribution.

7.2.3.1.1 After removal from their respective semi-VOST, cartridges were sealed in glass containers, identified with a QA control number, and dispatched to laboratories. To ensure prompt and undamaged delivery, all cartridge samples were hand-carried by courier.

7.2.3.1.2 Quartz-fiber filters were sealed in aluminum foil, two per wrapping, identified with a QA control number, and dispatched to laboratories. Samples designated for AWL were hand-carried by courier; samples designated for BCD were shipped by common carrier.

7.2.3.1.3 Quartz tissue paper filter and PUF filters used for dioxin sampling were sealed in BCD containers, identified with QA control numbers, and hand-carried to BCD by the test personnel who conducted the dioxin sampling.

7.2.3.1.4 Teflon™ and Nuclepore™ filters were sealed in aluminum foil, identified with a QA control number, and delivered to SNL staff for in-house assay, or transferred to a supporting laboratory for assay.

7.2.3.1.5 Bubblers, with their collection fluids, were given QA numbers and hand-carried by SNL staff to an SNL facility for assay.

7.2.3.1.6 Canister samples. After being assigned QA numbers, all 6-L and 0.85-L canisters were packaged in shock-resistant containers and shipped, via common carrier, to OGC for assay.

7.2.3.1.7 Other samples. Remaining samples were packaged in sealed containers and delivered to proper laboratories for assay.

7.2.3.2. Detection and Identification

7.2.3.2.1 Organics. The GC/MS was used to identify and quantify permanent gas and volatile organic species. Both SFC/MS and GC/MS were used to identify and quantify other organic species. SFC/MS and GC/MS procedures are outlined in Volume 3.

7.2.3.2.2 Elementals. XRF was used to determine elemental content. Procedures for this type assay are found in Volume 3.

7.2.3.3. Carbon Analysis. Pyrolysis/combustion techniques, described in Volume 3 were used in conducting carbon analysis.

7.2.3.4. TSP. Gravimetric analysis was used to determine TSP.

7.2.3.5. Assessment of carbon balance Method.

7.2.3.5.1 Comparison of cloud volume method and carbon balance method. The results of the sample analyses, together with the real-time data, were interpreted using both the cloud volume method (considering the volume of the chamber to be the cloud volume) and the carbon balance

method. EFs calculated by the cloud volume method were compared to those calculated by the carbon balance method. This comparison was conducted to determine whether, on subsequent field tests, the carbon balance method could be used exclusively for extrapolating cloud contents from sampling results, or whether it would have to be used in combination with other procedures.

7.2.3.5.2 BB volume was determined by interpretation of SF₆ tracer gas analysis results.

7.2.3.6. Assessment of Instrumental and Canister Sampling Techniques. Using analytical data developed both on-site and during chemical assay, the instrumental and canister sampling techniques used during this subtest were examined for their application to anticipated outdoor testing, mounted aboard a mobile aerial platform (such as the SNL FWAC).

7.2.3.7. Evaluation of the SFC/MS Results. SFC/MS results were examined by the PM and the TSC to determine if the SFC/MS analysis method was suitable for use as the principal separation and analysis technique during future OB/OD field trials. This examination included comparison of SFC/MS results with compounds and amounts known to be in spiked samples provided by EPA, and, where possible, with results of the GC/MS.

7.2.3.8. The degree to which specified dioxins were produced was determined by GC/MS analysis.

7.3. Test Findings

7.3.1. Carbon Mass Balance.

The available composite propellant carbon mass was estimated by two methods: (1) the amount released (calculation based on the composite propellant formulation; from carbon analysis of propellant samples by M-H-W Laboratories, Phoenix, Arizona) and (2) the amount measured (accounting for all carbon-containing products through analysis of aerosol and particulate samples taken during the trial). The mass of composite propellant burned was 448.41 g.

7.3.1.1. The percent of available carbon calculated from information on the formulation of the composite propellant was 11.41 percent. This fraction of 0.1141 was used as the fraction of carbon

in the burn fuel for calculation of EF of the products resulting from the burn. The percent carbon from the composite propellant has a lot of uncertainty due to the fact that it is a composite from multiple sources with a butyl binder. This uncertainty is apparent when examining the laboratory assay results from 3 samples in paragraph 7.3.1.2.

7.3.1.2. The M-H-W laboratories analysis of three composite propellant samples gave the following percentages for the elemental carbon:

Composite Propellant Sample #1:	21.26 percent carbon
Composite Propellant Sample #2:	12.67 percent carbon
Composite Propellant Sample #3:	17.15 percent carbon

The average percent carbon is 17.03 with a 95 percent confidence interval of 6.35 to 27.70 percent.

7.3.1.3. Analysis of the air samples taken during the trial provided an estimate of the total carbon mass based on the combined carbon mass contained in CO₂, CO, organic carbon, and the elemental carbon. The carbon contributed by CO₂ was estimated from the CO₂ concentration derived from real-time (continuously monitored) instrument sampling. Estimates immediately after detonation were derived by extrapolation of the fitted exponential curve (fit to the concentration data from the homogeneous period of sampling) to detonation time ($t = 0$). The carbon mass contributed from CO was estimated from the 6-L canister samples with extrapolation of the fitted exponential curve of concentration values to detonation time. The organic carbon and elemental carbon contributions were estimated by thermal analysis of a 1-cm² sample taken from the quartz-fiber filter of the semi-VOST. The analysis incorporated a two-step volatilization and combustion process to differentiate between volatile and elemental carbon on the filter. The mass of carbon contributed by these four sources as a result of the one composite burn conducted 16 February 1989 was 66.12 g. This carbon mass value is 29 percent greater than that calculated from the propellant formulation (51.16 g), and is 13 percent lower than the amount calculated using the carbon analysis data from M-H-W Laboratories (73.36 g). For purposes of calculating EF values, 51.16 g, based on data for the formulation of the composite propellant, was used.

7.3.2. Emission Factors.

The EFs of compounds and elements detected from the composite propellant burn were calculated by two methods; The carbon balance method and the second method, referred to as the cloud volume method, described in paragraph 5.3.2.1 and paragraph 5.3.2.3 respectively.

7.3.2.1. Real Time Continuously Monitored Gases. The concentration data used in computing EFs for CO₂, NO, NO₂ and SO₂ were those from the homogeneous sampling period (after 3 min of fanning). The resulting EFs, calculated by both methods, for these continuously monitored gases are in Table 7.2.

Table 7.2 EF for the Continuously Monitored Gases CO₂, NO, NO₂ and SO₂ from the Composite Propellant Burn, Calculated by the Carbon Balance and by the Cloud Volume Methods.

Method	Sample Source	Compound			
		CO ₂	NO	NO ₂	SO ₂
Carbon Balance	Direct	4.17 x 10 ⁻¹	4.16 x 10 ⁻³	0.00	1.12 x 10 ⁻⁴
	Indirect	4.18 x 10 ⁻¹	2.79 x 10 ⁻³	7.77 x 10 ⁻⁴	8.34 x 10 ⁻⁵
		4.18 x 10 ⁻¹	2.68 x 10 ⁻³	6.54 x 10 ⁻⁴	7.81 x 10 ⁻⁵
		4.17 x 10 ⁻¹	2.18 x 10 ⁻³	9.94 x 10 ⁻⁴	6.19 x 10 ⁻⁵
	Average	4.18 x 10 ⁻¹	2.95 x 10 ⁻³	6.06 x 10 ⁻⁴	8.38 x 10 ⁻⁵
Cloud Volume	Direct	5.40 x 10 ⁻¹	5.37 x 10 ⁻³	0.00	1.44 x 10 ⁻⁴
	Indirect	5.81 x 10 ⁻¹	3.88 x 10 ⁻³	1.08 x 10 ⁻³	1.16 x 10 ⁻⁴
		5.98 x 10 ⁻¹	3.85 x 10 ⁻³	9.36 x 10 ⁻⁴	1.12 x 10 ⁻⁴
		6.61 x 10 ⁻¹	3.45 x 10 ⁻³	1.57 x 10 ⁻³	9.80 x 10 ⁻⁵
	Average	5.95 x 10 ⁻¹	4.14 x 10 ⁻³	8.98 x 10 ⁻⁴	1.18 x 10 ⁻⁴

7.3.2.1.1 The cloud volume method EF's are all larger than the EFs from the carbon balance method for these continuously monitored gases. The ratio of the carbon balance method EF to the cloud volume method EF is approximately 0.7. A principal reason that this ratio is not closer to 1 is the difficulty in determining precisely the average amount of carbon in the fuel. The variability of the carbon content of the composite propellant burned in this test, as also noted from the M-H-W Laboratories' carbon analysis of three samples (range 12.67 to 21.26 percent), points out the need for a better characterization of the carbon fraction in the propellant when heterogeneous

mixtures are being studied. Because of this variability, calculated product EFs were based on the amount of available carbon from the estimated chemical formulation of the composite propellant.

7.3.2.2. Canister-Sampled CO₂, CO and Volatile Organics.

7.3.2.2.1 Samples of CO₂, CO, the volatile compounds (methane, acetylene, benzene, and a series of paraffins, olefins, non-benzene aromatics, and terpenes) were collected by 6-L canisters during the homogeneous period. The list of compounds considered within these groupings are outlined in Table 5.7. The EFs are in Table 7.3.

Table 7.3 EF for the Canister-Sampled CO₂, CO, and Volatile Organics from the Composite Propellant Burn, Calculated by the Carbon Balance and by the Cloud Volume Methods.

Method	Sample Source	Volatile Organics (Exotics)							
		Carbon Dioxide	Carbon Monoxide	Methane	Paraffins	Olefins	Non-Benzene Aromatics	Acetylene	Benzene
Carbon Balance	Direct	4.17 x 10 ⁻¹	3.52 x 10 ⁻⁴	0.00	6.37 x 10 ⁻⁶	1.51 x 10 ⁻⁵	7.86 x 10 ⁻⁶	5.10 x 10 ⁻⁶	4.46 x 10 ⁻⁶
		4.17 x 10 ⁻¹	3.20 x 10 ⁻⁴	0.00	2.08 x 10 ⁻⁵	1.56 x 10 ⁻⁵	6.41 x 10 ⁻⁶	2.29 x 10 ⁻⁶	5.04 x 10 ⁻⁶
		4.17 x 10 ⁻¹	3.01 x 10 ⁻⁴	0.00	1.03 x 10 ⁻⁶	8.76 x 10 ⁻⁶	8.50 x 10 ⁻⁶	2.06 x 10 ⁻⁶	5.15 x 10 ⁻⁶
		4.17 x 10 ⁻¹	3.12 x 10 ⁻⁴	3.19 x 10 ⁻⁵	6.49 x 10 ⁻⁷	9.08 x 10 ⁻⁶	1.10 x 10 ⁻⁵	1.30 x 10 ⁻⁶	6.49 x 10 ⁻⁶
		4.17 x 10 ⁻¹	2.86 x 10 ⁻⁴	6.56 x 10 ⁻⁵	8.24 x 10 ⁻⁷	1.67 x 10 ⁻⁶	1.63 x 10 ⁻⁵	0.00	7.09 x 10 ⁻⁶
	Average	4.17 x 10 ⁻¹	3.14 x 10 ⁻⁴	1.95 x 10 ⁻⁵	5.94 x 10 ⁻⁶	1.00 x 10 ⁻⁵	1.00 x 10 ⁻⁵	2.15 x 10 ⁻⁶	5.65 x 10 ⁻⁶
	Indirect	3.39 x 10 ⁻¹	2.68 x 10 ⁻⁴	0.00	4.99 x 10 ⁻⁶	2.82 x 10 ⁻⁵	1.89 x 10 ⁻⁶	1.38 x 10 ⁻⁶	1.82 x 10 ⁻⁴
		3.57 x 10 ⁻¹	2.80 x 10 ⁻⁴	0.00	1.12 x 10 ⁻⁶	4.71 x 10 ⁻⁶	4.04 x 10 ⁻⁶	4.49 x 10 ⁻⁷	2.11 x 10 ⁻⁵
		3.54 x 10 ⁻¹	2.71 x 10 ⁻⁴	6.24 x 10 ⁻⁵	3.94 x 10 ⁻⁶	2.96 x 10 ⁻⁶	8.87 x 10 ⁻⁶	0.00	1.61 x 10 ⁻⁵
	Average	3.50 x 10 ⁻¹	2.73 x 10 ⁻⁴	2.08 x 10 ⁻⁵	3.35 x 10 ⁻⁶	1.20 x 10 ⁻⁵	4.93 x 10 ⁻⁶	6.08 x 10 ⁻⁷	7.31 x 10 ⁻⁵
Cloud Volume	Direct	4.32 x 10 ⁻¹	3.65 x 10 ⁻⁴	0.00	6.59 x 10 ⁻⁶	1.56 x 10 ⁻⁵	8.13 x 10 ⁻⁶	5.27 x 10 ⁻⁶	4.61 x 10 ⁻⁶
		4.66 x 10 ⁻¹	3.57 x 10 ⁻⁴	0.00	2.33 x 10 ⁻⁵	1.74 x 10 ⁻⁵	7.16 x 10 ⁻⁶	2.56 x 10 ⁻⁶	5.63 x 10 ⁻⁶
		5.08 x 10 ⁻¹	3.66 x 10 ⁻⁴	0.00	1.25 x 10 ⁻⁶	1.07 x 10 ⁻⁵	1.03 x 10 ⁻⁵	2.51 x 10 ⁻⁶	6.27 x 10 ⁻⁶
		5.07 x 10 ⁻¹	3.79 x 10 ⁻⁴	3.87 x 10 ⁻⁵	7.88 x 10 ⁻⁷	1.10 x 10 ⁻⁵	1.34 x 10 ⁻⁵	1.58 x 10 ⁻⁶	7.88 x 10 ⁻⁶
		5.09 x 10 ⁻¹	3.49 x 10 ⁻⁴	7.99 x 10 ⁻⁵	1.02 x 10 ⁻⁶	2.03 x 10 ⁻⁶	1.98 x 10 ⁻⁵	0.00	8.63 x 10 ⁻⁶
	Average	4.84 x 10 ⁻¹	3.63 x 10 ⁻⁴	2.37 x 10 ⁻⁵	6.58 x 10 ⁻⁶	1.13 x 10 ⁻⁵	1.18 x 10 ⁻⁵	2.38 x 10 ⁻⁶	6.60 x 10 ⁻⁶
	Indirect	4.73 x 10 ⁻¹	3.73 x 10 ⁻⁴	0.00	6.95 x 10 ⁻⁶	3.93 x 10 ⁻⁵	2.64 x 10 ⁻⁶	1.92 x 10 ⁻⁶	2.54 x 10 ⁻⁴
		5.12 x 10 ⁻¹	4.01 x 10 ⁻⁴	0.00	1.61 x 10 ⁻⁶	6.75 x 10 ⁻⁶	5.78 x 10 ⁻⁶	6.42 x 10 ⁻⁷	3.02 x 10 ⁻⁵
		5.61 x 10 ⁻¹	4.28 x 10 ⁻⁴	9.88 x 10 ⁻⁵	6.24 x 10 ⁻⁶	4.68 x 10 ⁻⁶	1.40 x 10 ⁻⁵	0.00	2.55 x 10 ⁻⁵
	Average	5.15 x 10 ⁻¹	4.01 x 10 ⁻⁴	3.29 x 10 ⁻⁵	4.93 x 10 ⁻⁶	1.69 x 10 ⁻⁵	7.48 x 10 ⁻⁶	8.53 x 10 ⁻⁷	1.03 x 10 ⁻⁴

7.3.2.3. A comparison of the mean EF values calculated for 6-L canister-sampled CO₂ and CO from the chamber (direct sampling), showed a statistical difference between values derived from the carbon balance method and those from the cloud volume method; however, no differences were found between methods for the EF's calculated for any of the other compounds or groups of compounds.

7.3.2.4. The range of EFs noted for the volatile hydrocarbons are given in Table 7.4.

Table 7.4 EF Range for Volatile Hydrocarbons.

Compound	Direct	Indirect
Methane	0 to 7.99×10^{-5}	0 to 9.88×10^{-5}
Paraffins	6.49×10^{-7} to 2.33×10^{-5}	1.12×10^{-6} to 6.95×10^{-5}
Acetylene	0 to 5.27×10^{-5}	0 to 1.92×10^{-6}
Olefins	1.67×10^{-6} to 1.74×10^{-5}	2.96×10^{-6} to 3.93×10^{-5}
Benzene	4.46×10^{-6} to 8.63×10^{-6}	1.61×10^{-5} to 3.02×10^{-5}
Nonbenzene aromatics	6.41×10^{-6} to 1.98×10^{-5}	1.89×10^{-6} to 1.40×10^{-5}

7.3.2.4.1 No EFs are shown for the terpene groups of compounds because they were not detected in the composite propellant burn trial samples.

7.3.2.4.2 Using the largest calculated EF's the maximum quantities of emissions from each category of volatile organics expected or released from a composite propellant burn were calculated and are shown in Table 7.5.

Table 7.5 Maximum Quantities of Emissions from each Category of Volatile Organics Released from a Composite Propellant Burn.

Compound	Source Strength (1-lb burn) (lb)	Source Strength (2000-lb burn) (lb/g)
Methane	9.88×10^{-5}	0.198/89.9
Paraffins	6.95×10^{-5}	0.139/63.1
Acetylene	5.27×10^{-5}	0.105/47.7
Olefins	3.93×10^{-5}	0.0786/35.7
Benzene	3.02×10^{-5}	0.0604/27.4
Nonbenzene aromatics	1.98×10^{-5}	0.0396/18.0
Totals	3.10×10^{-4}	0.621/281

7.3.2.5. Semivolatile Organics.

7.3.2.5.1 The test design provided data for calculation of EFs for semivolatile organics from the direct semi-VOST, the indirect/inhomogeneous semi-VOST, and the indirect/homogeneous semi-VOST. The sampling train consisted of a 10-cm diameter quartz-fiber filter followed by a glass cylinder packed with 65 g of XAD resin, followed by a second glass cylinder packed with 20 g of XAD resin. The net amounts of semivolatile organic compounds detected, calculated by summing the amounts found in each of the three components of the sampling train after correcting for background, were converted to EFs for each of the following compounds:

2,6-Dinitrotoluene
 4-Nitrophenol
 2,4-Dinitrotoluene
 2-Nitronaphthalene
 1-Nitropyrene
 Phenol
 Dibenzofuran
 N-Nitrosodiphenylamine
 Naphthalene

7.3.2.5.2 The following compounds, although looked for, were never found:

2,4,6-Trinitrotoluene
1,6-Dinitropyrene
2-Naphthalamine
Diphenylamine
Benz[c]acridine
Benz[a]anthracene
Benzo[a]pyrene
Dibenz[a,h]anthracene
2,2-Methylene bis(4-methyl)-6-t-butylphenol
Phenyl di-isodecyl phosphite
5-ethyl-1,3-diglycidyl-5-methyl hydantoin diepoxide
Diethylenetriamine
Dioctyl sebacate
Isophorone di-isocyanate

7.3.2.5.3 The 23 target analyte organic compounds cited in the two lists above are those which were searched for and, when found, measured by GC/MS and/or SFC/MS selected-ion monitoring techniques. Basically, the target analyte list consisted of: expected organic components of the composite propellant which may be present from the burning of the composite propellant in the chamber; and combustion product compounds which, if present in high concentration, would be of environmental concern.

7.3.2.5.4 The analysis results for each of the nine compounds that were detected at one time or another during the analyses performed are discussed separately in subparagraphs below. These discussions include a value for the EF mean and its 95 percent confidence interval. In computing this mean, all pertinent analysis data are combined for the given species; thus any variation contributed by differences in EF calculated using data from different trial days, sampler source (direct, indirect-nonhomogeneous, and indirect-homogeneous) analytical lab, or individual instrument is included as a part of the overall EF error variation. Missing cells in the tables of calculated EF values are due to the fact that a number of the quartz-fiber filters from the

semi-VOST were only assayed for organic carbon and elemental carbon and, thus, no SFC/MS or GC/MS assays were performed on these filters which would yield EF values.

7.3.2.5.5 Analysis Results for Each Semivolatile Organic Compound.

a. 2,6-Dinitrotoluene. The two estimates of EFs are 0 for the indirect-homogeneous sampling and 3.72×10^{-9} for the direct sampling. Using the 3.72×10^{-9} value and assuming a one-pound composite propellant burn, no more than 3.72-billionths of a pound of 2,6-dinitrotoluene is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than eight millionths of a pound ((0.0000074 lb) or 0.00336 g) of 2,6-dinitrotoluene would result.

b. 4-Nitrophenol. The two estimates of EFs are 4.08×10^{-7} for the indirect-homogeneous sampling and 4.96×10^{-8} for the direct sampling. Using the 4.08×10^{-7} value and assuming a 1-lb composite propellant burn, no more than 4.08-billionths of a pound of 4-nitrophenol is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than nine ten-thousandths of a pound ((0.000816 lb) or 0.370 g) of 4-nitrophenol would result.

c. 2,4-Dinitrotoluene. The two estimates of EFs are 0 for the indirect-homogeneous sampling and 1.04×10^{-8} for the direct sampling. Using the 1.04×10^{-8} value and assuming a one-pound composite propellant burn, no more than 10.4-billionths of a pound of 2,4-dinitrotoluene is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than twenty-one millionths of a pound ((0.0000208 lb) or 0.00943 g) of 2,4-dinitrotoluene would result.

d. 2-Nitronaphthalene. The two estimates of EFs are 1.26×10^{-8} for the indirect-homogeneous sampling and 2.03×10^{-8} for the direct sampling. Using the 2.03×10^{-8} value and assuming a one-pound composite propellant burn, one could expect no more than 20.3-billionths of a pound of 2-nitronaphthalene to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than fifty-millionths of a pound ((0.0000406 lb) or 0.0184 g) of 2-nitronaphthalene would result.

e. 1-Nitropyrene. The only estimate of EF is 1.98×10^{-8} for the direct sampling. Using the 1.98×10^{-8} value and assuming a one-pound composite propellant burn, no more than 19.8-millionths of a pound of 1-nitropyrene is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than forty-millionths of a pound ((0.0000396 lb) or 0.018 g) of 1-nitropyrene would result.

f. Phenol. The estimate of EF are is 3.78×10^{-6} from the direct sampling data. Using the 3.78×10^{-6} value and assuming a one-pound composite propellant burn, no more than 3.78-millionths of a pound of phenol is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than eight one-thousandths of a pound ((0.00756 lb) or 3.43 g) of phenol would result.

g. Dibenzofuran. The two estimates of EFs are 2.79×10^{-7} for the indirect-homogeneous sampling and 1.17×10^{-7} for the direct sampling. Using the 2.79×10^{-7} value and assuming a one-pound composite propellant burn, no more than 2.79-billionths of a pound of dibenzofuran is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than six ten-thousandths of a pound ((0.000558 lb) or 0.253 g) of dibenzofuran would result.

h. N-Nitrosodiphenylamine. The two estimates of EFs are 0 for the indirect-homogeneous sampling and 3.45×10^{-8} for the direct sampling. Using the 3.45×10^{-8} value and assuming a one-pound composite propellant burn, no more than 34.5-billionths of a pound of N-nitrosodiphenylamine is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than one ten-thousandths of a pound ((0.000069 lb) or 0.0313 g) of N-nitrosodiphenylamine would result.

i. Naphthalene. The EF from the indirect-homogeneous sampling data is 1.44×10^{-6} . Using the 1.44×10^{-6} value, and assuming a one-pound composite propellant burn, no more than 1.44-millionths of a pound of naphthalene is expected to be produced as a combustion product of the composite propellant. For a one-ton (2,000-lb) composite propellant burn, provided linear scaling applies, less than three one-thousandths of a pound ((0.00288 lb) or 1.31 g) of naphthalene would result.

7.3.2.6. Elements. Results from XRF analysis of filter samples obtained from direct sampling during the composite propellant trial of 16 February 1989 were used in calculating the EFs presented in Table 7.6. A number of metals and nonmetals were seen in these samples, but only aluminum and iron are present in the propellant formulation; neither of these metals are on the target analyte list. Because the metals present in the propellant (Al and Fe) were not considered to be environmentally important and the other metals and nonmetals found in the atmosphere had to be derived from chamber surfaces or contamination from earlier SNL experiments EFs for those metals have no practical significance.

Table 7.6 EF for Target Elements, Aluminum and Iron Found on Particles from the Filter Samples, Direct BB Sampling of the Composite Propellant Burn.

Element	Emission Factor
Aluminum	1.33×10^{-4}
Chromium ^a	4.77×10^{-6}
Iron	6.04×10^{-4}
Nickel ^a	1.59×10^{-6}
Arsenic ^a	0.00
Lead ^a	9.39×10^{-5}
Cadmium ^a	0.00
Antimony ^a	4.77×10^{-6}
Barium ^a	1.59×10^{-7}

^aElement is on target analyte list, but not present in the composite propellant (fuel).

7.3.2.7. Ammonia. Solvent-filled bubblers were used during the BB trials to collect NH₃. No NH₃ was detected in the samples from the composite propellant burn trial.

7.3.2.8. Hydrogen Cyanide. Solvent-filled bubblers were used to collect HCN. HCN was at nondetectable levels (approximately 0.080 mg/m³) in samples from the composite propellant burn trial.

7.3.2.9. Hydrogen Chloride. Solvent-filled bubblers aspirated for the determination of HCl yielded an EF of 9.43×10^{-3} . This corresponds to 188.6 lbs (85.5 kg) per ton of this composite propellant. If all of the ammonium perchlorate assumed to be present in the propellant were converted to HCl, 527.6 lbs per ton would have been produced. An HCl EF of 4.79×10^{-4} was obtained by analyzing

XRF data on chlorine found in compounds retained as particles on the direct filter sample. This corresponds to 0.958 lbs (0.43 g) of chlorine in solid compounds per ton of composite propellant. Thus, only about 36 percent of the chlorine available in the unburned propellant fuel is being detected as HCl using the bubbler sampling method. Chlorine/HCl sampling procedures will be reviewed and revisions made, as necessary, for later composite burn phases of the OB/OD program.

7.3.2.10. Polychlorinated Dibenzodioxins (PCDDs).

High-volume air samplers (283 L/min) were used to take duplicate samples on quartz fiber filters backed up by pre-cleaned PUF-filled cartridges. The resulting samples were extracted and the separate solutions subjected to the special analytical procedure required for PCDDs and for polychlorinated dibenzofurans (PCDFs). The analysis included the total hepta-, hexa-, penta-, and tetra-CDD and CDF congeners, as well as octa-, and 2,3,7,8-tetra-CDD and CDF. Two dibenzodioxins were found in some of the composite propellant burn samples. A net concentration of $8.6 \times 10^{-6} \mu\text{g}/\text{m}^3$ octachlorodibenzodioxin (OCTA-CDD) was found in the BB air during the trial. However, OCTA-CDD is a common contaminant of many sample matrices, and the toxicity of this compound is less than 0.0001 the toxicity of 2,3,7,8-tetra-CDD. Hepta-chlorodibenzodioxin (HEPTA-CDD) was found in only one set of air samples, but not in the duplicate set, giving an average concentration of $1.3 \times 10^{-6} \mu\text{g}/\text{m}^3$ within the BB during the trial. The toxicity of HEPTA-CDD is 0.00001 that of 2,3,7,8-tetra-CDD. Taking into account relative toxicities, the total PCDD content corresponds to less than 2 ng ($2 \times 10^{-3} \mu\text{g}$) of 2,3,7,8-tetra-CDD per metric ton (1000 kg) of composite propellant. These results indicated a "clean" burn with respect to dioxin formation.

7.3.2.11. Polychlorinated Dibenzofurans.

The same samples used for analysis of PCDD analyses were also analyzed for PCDFs. The sums of the concentrations of the PCDF species averaged $7.5 \times 10^{-3} \mu\text{g}/\text{m}^3$ in the duplicate samples. The PCDFs were found on the filters only; thus, they were associated with particulate matter. Taking into account the relative toxicity of the congeners, this concentration in the BB corresponds to a yield of 3.3 μg of 2,3,7,8-tetra-CDF per metric ton of composite propellant.

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SECTION 8. FOAM-ATTENUATED TRINITROTOLUENE DETONATION

8.1. Objective

To assess the ability of commercially available firefighting foam to serve as a surrogate for soil as a blast-mitigant in chamber detonation trials.

8.2. Test Procedure

8.2.1. Data Required

8.2.1.1. Video coverage from t-1 to t+35 min by a standard-speed camera inside and outside the test chamber.

8.2.1.2. HS video coverage inside the test chamber from t-1 to t+10 min.

8.2.1.3. Particle Size Distribution.

8.2.1.3.1. One APS particle size distribution determination per minute, from t-45 to t+35 min.

8.2.1.3.2. One DMPS particle size distribution on every 5 min, from t-45 to t+35 min.

8.2.1.4. Analog data (5-s intervals), from t-45 to t+35 min, from the following instruments:

8.2.1.4.1. Nephelometer.

8.2.1.4.2. CO₂ analyzer (± 2 ppm).

8.2.1.4.3. CO analyzer (± 0.1 ppm).

8.2.1.4.4. NO_x analyzer (± 5 ppb).

8.2.1.4.5. SO₂ analyzer (± 2 ppb).

8.2.1.4.6. O₃ analyzer (± 2 ppb).

8.2.1.4.7. GC-PID analysis for organic compounds (± 1 ppm).

8.2.1.5. Real-Time Test Conditions:

8.2.1.5.1. Differential pressure between the interior and exterior of the BB (± 6 mm of H₂O);

8.2.1.5.2. BB temperature ($\pm 0.5^\circ\text{C}$);

8.2.1.5.3. BB dew/frost point ($\pm 0.5^\circ\text{C}$);

8.2.1.5.4. Bag sampler valve position;

8.2.1.5.5. Gas analyzer valve position.

8.2.1.6. Analog data (5-s intervals) from the following instruments on indirect bag samples taken at $t-30$, $t+3$, and $t+15$ min while the air bag was being pumped down:

8.2.1.6.1. CO₂ analyzer (± 0.1 ppm);

8.2.1.6.2. CO analyzer (± 0.1 ppm).

8.2.1.7. SFC/MS analysis of fresh and post-detonation foam samples.

8.2.1.8. XRF analysis for elementals from samples collected by Teflon™ filters sampling from the 1.5-m³ Velostat™ bag at $t-45$, $t+5$, $t+15$, and $t+30$ min.

8.2.1.9. SEM analysis of samples collected by Nuclepore™ filters sampling from the 1.5-m³ Velostat™ bag at t-45, t+5, t+15, and t+30 min.

8.2.1.10. Analyses for THC, CH₄, C₂-C₁₀HC, CO, CO₂, and H₂ drawn from inside the chamber by 6-L canisters at t-45, t+2, t+14, t+18, t+23, and t+30.

8.2.1.11. Analyses for THC, CH₄, C₂-C₁₀HC, CO, CO₂, and H₂ drawn from the 1.5-m³ Velostat™ bag by 6-L canisters at t-45, t+4, t+15, and t+30.

8.2.2. Data Acquisition Procedures

8.2.2.1. At t-55 min, the 1.5-m³ Velostat™ bag drew air from the chamber through the sampling tube. Contents of the bag were sampled by real-time analyzers and by a 6-L canister.

8.2.2.2. A heavy-duty plastic cage was prepared to contain the foam during the subtest until the TNT charge was detonated. The cage, made of 5-cm mesh approximately 183 cm in diameter and 92 cm high, surrounded the detonation stand used throughout the test. Glad Cling Wrap™ liner sheets were draped from outside the upper part of the cage, over the top, down the inner cage wall, and across the bottom center. The liner sheets overlapped 5 cm on the sides and 30 cm on the ends to prevent the foam from seeping outside the cage.

8.2.2.3. Two shallow, 4.6-L aluminum sampling pans were placed inside the cage against opposite walls.

8.2.2.4. After a 221.6gram TNT block was suspended in the detonation stand and wired for detonation, nonessential personnel left the test chamber and foam generation began. The foam generator was operated by a single technician and produced foam with an expansion ratio (ER) of 130:1. (The ER is the ratio of the volume of expanded foam to the volume of water and concentrate used.) Foam generation continued for approximately 8 min. until the TNT charge was covered and the foam reached the upper edge of the cage wall. The ratio of the diameter of generated foam to that for the explosive charge (17:1) was the same as had proven effective during prior experiments conducted at the Naval Ordnance Station, Indianhead, Maryland, and at SNL in

previous tests. Components of the foam used were: (1) a glycol ether, (2) a 5-carbon alcohol, (3) xanthan biopolymer, (4) formaldehyde, (5) a sulfonate surfactant, and (6) fatty alcohols (C_{12} - C_{14}).

8.2.2.5. Real-time instruments, running continuously throughout the test, sampled chamber air before and during this subtest.

8.2.2.6. The following background samples were drawn by 6-L canisters:

8.2.2.6.1. Indirect sample, from the chamber via the 1.5-m³ Velostat™ bag, at t-55 min.

8.2.2.6.2. Direct sample, from the chamber, at t-5 min (following completion of foam generation).

8.2.2.6.3. Direct sample, outside the BB, at t-58, t+3, and t+14 min.

8.2.2.7. The following post-detonation air samples were drawn by 6-L canister from inside the chamber:

8.2.2.7.1. Indirect sample at t+4, t+16, and t+32.

8.2.2.7.2. Direct sample at t+2, t+14, t+18, t+23, and t+29 min. Two samples were drawn at t+2 min; one from 2 m above the floor, and the other from the top of the chamber.

8.2.2.8. Fresh foam samples were taken from inside the cage before the detonation and were placed in sample jars, which were then labeled and sealed.

8.2.2.9. Post-detonation foam samples were collected from sample pans and scooped from the floor and placed in individual sample jars, which were then labeled and sealed.

8.2.2.10. The pressure differential between inside and outside the chamber was constantly monitored and adjusted to ensure adequate flow rates into the Velostat™ bag.

8.2.3. Analytical Procedures

8.2.3.1. Sample Distribution

8.2.3.1.1. Canisters were packed in shock-resistant containers for shipment via common carrier, to OGC for assay of volatile organics by GC.

8.2.3.1.2. Filter samples were packaged in sealed containers and delivered to respective laboratories for element and particle analyses.

8.2.3.2. Detection and Identification

8.2.3.2.1. Organics. SFC/MS and GC/MS were used to detect and identify organic species. Both AWL and BCD used GC/MS for semivolatile organic compound analyses; AWL also used the SFC/MS. MS was used by both laboratories. OGC used GC to identify and quantitate volatile organics.

8.2.3.2.2. Elementals. XRF was used to determine elemental content.

8.3. Test Findings

8.3.1. Carbon Mass Balance.

The carbon mass was determined from the TNT formulation ($C_7 H_5 N_3 O_6$), from independent elemental analysis laboratory (M-H-W Laboratories) analysis, and from results of analysis of the aerosol samples taken during the trial.

8.3.1.1. The theoretical amount of carbon is 37.01 percent.

8.3.1.2. The independent laboratory analysis of two TNT samples give the following results for the elemental analysis:

Sample #0043: 37.68 percent carbon

Sample #0044: 37.53 percent carbon

These samples were described as scrapings from the corners of the pressed block. These samples may not be representative of the cast TNT block, because surface scrapings are subject to ambient air exposure, and some discoloring had occurred on some blocks.

8.3.1.3. The air samples taken during the trial provided an estimate of the total carbon mass of carbon based on the carbon contained in CO_2 and CO. The carbon from CO_2 was estimated from the real-time (continuously monitored) instrument sampling for CO_2 , with extrapolation of the fitted exponential curve (fit to the data from the homogeneous period of sampling) to detonation time ($t = 0$). The carbon from CO was estimated from the 6-L canister sampling, with extrapolation of the fitted exponential curve to detonation time. The organic carbon and elemental carbon were not estimated because no quartz-fiber filter samples were taken. The mass of carbon recovered from the one foam-attenuated trial was 26.85 g. This represented only 33 percent of the theoretical fuel carbon (82.00 g) and 32 percent of the elemental analysis estimate of carbon (83.31 g).

8.3.2. Emission Factors.

The EFs were calculated by two methods; the carbon balance method, and the cloud volume method, are described in paragraphs 5.3.2.1 and 5.3.2.2 respectively.

8.3.3. Real Time Continuously Monitored Gases.

CO_2 , NO, NO_2 , and SO_2 . The EFs shown in Table 8.1 are all from a homogeneous sampling period.

Table 8.1 EF for the Continuously Monitored Gases CO₂, NO, NO₂, and SO₂ from the Foam-Attenuated TNT Detonation, Calculated by the Carbon Balance and by the Cloud Volume Methods.

Method	Sample Source	Compound			
		CO ₂	NO	NO ₂	SO ₂
Carbon Balance	Direct	1.22	4.95 x 10 ⁻³	3.86 x 10 ⁻³	1.10 x 10 ⁻⁴
	Indirect	1.24	4.15 x 10 ⁻³	4.10 x 10 ⁻³	8.46 x 10 ⁻⁵
		1.24	4.08 x 10 ⁻³	5.29 x 10 ⁻³	1.11 x 10 ⁻⁴
		1.24	3.50 x 10 ⁻³	7.63 x 10 ⁻³	1.11 x 10 ⁻⁴
	Average	1.23	4.17 x 10 ⁻³	5.22 x 10 ⁻³	1.04 x 10 ⁻⁴
Cloud Volume	Direct	3.99 x 10 ⁻¹	1.62 x 10 ⁻³	1.26 x 10 ⁻³	3.60 x 10 ⁻⁵
	Indirect	4.33 x 10 ⁻¹	1.45 x 10 ⁻³	1.44 x 10 ⁻³	2.96 x 10 ⁻⁵
		4.62 x 10 ⁻¹	1.52 x 10 ⁻³	1.98 x 10 ⁻³	4.16 x 10 ⁻⁵
		4.71 x 10 ⁻¹	1.33 x 10 ⁻³	2.90 x 10 ⁻³	4.20 x 10 ⁻⁵
	Average	4.41 x 10 ⁻¹	1.48 x 10 ⁻³	1.89 x 10 ⁻³	3.73 x 10 ⁻⁵

8.3.3.1. Comparison of the EF means between the carbon balance method and the cloud volume method showed that differences exist for CO₂, NO, NO₂, and SO₂. The differences are traceable to the inability of the air sampling to recover all the carbon in the foam-attenuated trial. Since all carbon and carbon compounds are not air borne the BB sampling could not establish a mass balance of carbon. As shown in paragraph 8.3.1.3, approximately 33 percent of the total carbon mass was found; therefore the carbon balance method will overestimate the mass of a species from the detonation by threefold.

8.3.4. Canister-Sampled CO₂, CO and Volatile Organics.

8.3.4.1. Samples for CO₂, CO, and the volatile compounds (methane, acetylene, and benzene, and a series of paraffins, olefins, nonbenzene aromatics, and terpenes (terpenes never found)) were collected by the 6-L canister during the homogeneous period. The EFs for the compounds and groups of compounds are in Table 8.2.

Table 8.2 EF for the Canister Sampled CO₂, CO, and the Volatile Organics from the Foam-Attenuated TNT Detonation, Calculated by the Carbon Balance and by the Cloud Volume Methods.

Method	Sample Source	Carbon Dioxide	Carbon Monoxide	Volatile Organics					
				Methane	Paraffins	Olefins	Non-Benzene Aromatics	Acetylene	Benzene
Carbon Balance	Direct	1.21	9.29 x 10 ⁻²	3.50 x 10 ⁻⁴	1.20 x 10 ⁻⁴	4.33 x 10 ⁻³	4.85 x 10 ⁻⁴	1.26 x 10 ⁻³	1.58 x 10 ⁻⁴
		1.21	9.15 x 10 ⁻²	4.11 x 10 ⁻³	2.10 x 10 ⁻³	4.81 x 10 ⁻⁴	4.07 x 10 ⁻⁴	1.18 x 10 ⁻³	1.64 x 10 ⁻⁴
		1.21	9.04 x 10 ⁻²	4.08 x 10 ⁻⁴	3.45 x 10 ⁻⁴	4.52 x 10 ⁻³	4.34 x 10 ⁻⁴	1.28 x 10 ⁻³	1.69 x 10 ⁻⁴
		1.21	9.35 x 10 ⁻²	3.65 x 10 ⁻⁴	4.40 x 10 ⁻³	4.61 x 10 ⁻³	5.77 x 10 ⁻⁴	1.29 x 10 ⁻³	1.65 x 10 ⁻⁴
		1.20	9.90 x 10 ⁻²	3.95 x 10 ⁻⁴	4.70 x 10 ⁻³	4.84 x 10 ⁻³	5.25 x 10 ⁻⁴	1.36 x 10 ⁻³	1.69 x 10 ⁻⁴
	Average	1.21	9.35 x 10 ⁻²	3.86 x 10 ⁻⁴	1.16 x 10 ⁻⁴	4.62 x 10 ⁻³	4.86 x 10 ⁻⁴	1.28 x 10 ⁻³	1.65 x 10 ⁻⁴
	Indirect	1.55	9.59 x 10 ⁻²	4.07 x 10 ⁻⁴	4.98 x 10 ⁻³	4.79 x 10 ⁻³	2.70 x 10 ⁻⁴	1.21 x 10 ⁻³	1.67 x 10 ⁻⁴
		0.635	5.92 x 10 ⁻²	2.47 x 10 ⁻⁴	3.35 x 10 ⁻³	2.69 x 10 ⁻³	1.79 x 10 ⁻⁴	9.28 x 10 ⁻⁴	1.10 x 10 ⁻⁴
		0.982	7.39 x 10 ⁻²	3.13 x 10 ⁻⁴	1.69 x 10 ⁻³	3.92 x 10 ⁻³	1.94 x 10 ⁻⁴	9.42 x 10 ⁻⁴	1.10 x 10 ⁻⁴
	Average	1.06	7.64 x 10 ⁻²	3.22 x 10 ⁻⁴	3.34 x 10 ⁻³	3.80 x 10 ⁻³	2.14 x 10 ⁻⁴	1.03 x 10 ⁻³	1.29 x 10 ⁻⁴
Cloud Volume	Direct	0.363	2.79 x 10 ⁻²	1.05 x 10 ⁻⁴	3.61 x 10 ⁻³	1.30 x 10 ⁻³	1.46 x 10 ⁻⁴	3.78 x 10 ⁻⁴	4.73 x 10 ⁻⁵
		0.371	2.81 x 10 ⁻²	1.26 x 10 ⁻⁴	6.44 x 10 ⁻⁶	1.47 x 10 ⁻³	1.25 x 10 ⁻⁴	3.62 x 10 ⁻⁴	5.02 x 10 ⁻⁵
		0.387	2.88 x 10 ⁻²	1.30 x 10 ⁻⁴	1.10 x 10 ⁻⁴	1.44 x 10 ⁻³	1.38 x 10 ⁻⁴	4.08 x 10 ⁻⁴	5.39 x 10 ⁻⁵
		0.361	2.79 x 10 ⁻²	1.09 x 10 ⁻⁴	1.32 x 10 ⁻³	1.38 x 10 ⁻³	1.72 x 10 ⁻⁴	3.86 x 10 ⁻⁴	4.93 x 10 ⁻⁵
		0.337	2.78 x 10 ⁻²	1.11 x 10 ⁻⁴	1.32 x 10 ⁻³	1.36 x 10 ⁻³	1.47 x 10 ⁻⁴	3.82 x 10 ⁻⁴	4.73 x 10 ⁻⁵
	Average	0.364	2.81 x 10 ⁻²	1.16 x 10 ⁻⁴	3.58 x 10 ⁻³	1.39 x 10 ⁻³	1.46 x 10 ⁻⁴	3.83 x 10 ⁻⁴	4.96 x 10 ⁻⁵
	Indirect	0.428	2.64 x 10 ⁻²	1.12 x 10 ⁻⁴	1.37 x 10 ⁻³	1.32 x 10 ⁻³	7.44 x 10 ⁻⁵	3.35 x 10 ⁻⁴	4.61 x 10 ⁻⁵
		0.301	2.81 x 10 ⁻²	1.17 x 10 ⁻⁴	1.59 x 10 ⁻³	1.28 x 10 ⁻³	8.48 x 10 ⁻⁵	4.40 x 10 ⁻⁴	5.20 x 10 ⁻⁵
		0.373	2.81 x 10 ⁻²	1.19 x 10 ⁻⁴	6.42 x 10 ⁻⁶	1.49 x 10 ⁻³	7.39 x 10 ⁻⁵	3.58 x 10 ⁻⁴	4.18 x 10 ⁻⁵
	Average	0.367	2.75 x 10 ⁻²	1.16 x 10 ⁻⁴	1.20 x 10 ⁻³	1.36 x 10 ⁻³	7.77 x 10 ⁻⁵	3.78 x 10 ⁻⁴	4.66 x 10 ⁻⁵

8.3.4.2. A statistical comparison of the carbon balance method and cloud volume method was not made because a carbon balance was not achieved (the carbon balance method does not yield equivalency in emission factors). All EFs from the carbon balance method yield approximately three times the mass of species that results from the cloud volume method. Failure to recover all the carbon results in an overestimation of the amount of a given species. Using the largest carbon balance EF from the volatile organics, 0.00484 (4.84×10^{-3}) and assuming a one-pound TNT foam-attenuated detonation, no more than five one-thousandths of a pound of olefins is expected to be produced as a combustion product or released as a contaminant.

8.3.5. Semivolatile Organics, Elementals, Ammonia, and Hydrogen Cyanide.

There was no sampling for these species.

8.4. Technical Assessment

8.4.1. The foam did not sufficiently attenuate detonation blast effects to prevent the plastic cage from being ruptured, or aluminum sampling pans from being distorted.

8.4.2. After detonation, foam residue on the floor within a 2-m radius of the enclosure center had changed colors, ranging from its original neutral-white, to a medium-gray to pitch black.

8.4.3. One sampling pan was blown towards the BB door and overturned. It was ultimately used to scoop foam off the aluminum plates directly below the detonation point. The other pan was blown toward the opposite wall and contained foam residue, which was sampled and sent to AVL for assay.

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APPENDIX A. CONSOLIDATED ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
AEHA	U.S. Army Environmental Hygiene Agency, Aberdeen Proving Ground, Maryland
AFB	Air Force Base
AMC	U.S. Army Materiel Command, Alexandria, Virginia
AMCCOM	U.S. Army Armament, Munitions and Chemical Command, Rock Island, Illinois
amino-PAH	aminopolycyclic aromatic hydrocarbons
ANOVA	analysis of variance
AP	ammonium perchlorate
APS	aerodynamic particle sizer
ASASP	active scattering aerosol spectrometer probe
AWL	Alpine West Laboratories, Provo, Utah
BB	BangBox
BCD	Battelle Columbus Division, Columbus, Ohio
BD	target analyte not found in concentrations above detection limits
BYU	Brigham Young University, Provo, Utah
CAA	Clean Air Act
CDD	chlorinated dibenzodioxin
CDF	chlorinated dibenzofuran
CI-SIM	chemical ionization, selective-ion monitoring
CSI	Columbia Scientific Instruments
C · V	concentration times cloud volume method
CWA	Clean Water Act
DMC	Data Management Center
DMPS	differential mobility particle sizer
DoD	Department of Defense
DPG	U.S. Army Dugway Proving Ground, Dugway, Utah
EC	electron capture <i>or</i> elemental carbon
ECD	electron capture detector
EDAX	energy-dispersive X-ray analysis
EER	Energy and Environmental Research Corporation, Irvine, California

EF	emission factor(s)
EI	electron impact
EI-MS	mass spectrometer used in the electron impact ionization mode
EI/MS	electron impact ionization/ mass spectrometry
EIS	environmental impact statement
ELI	Environmental Labs, Incorporated, Provo, Utah
EOD	explosive ordnance disposal
EPA	U.S. Environmental Protection Agency
EPO	Environmental Protection Office, U.S. Army Dugway Proving Ground. Dugway, Utah
ER	expansion ratio
FID	flame ionization detector
FSSP	forward scattering spectrometer probe
FTIR	Fourier Transform Infrared Spectrometry
FWAC	fixed-wing aircraft
GC	gas chromatograph(y)
GC-ECD	gas chromatography with an electron capture detector
GC-FID	gas chromatography with a flame ionization detector
GC/MS	gas chromatography-mass spectrometry
GLP	good laboratory practices
HE	high explosive
HMX	octamethylenehexanitramine
HNBB	hexanitrobibenzyl
HRGC/HRMS	combined capillary column gas chromatography/high resolution mass spectrometry
HS	high-speed
LASD	Los Angeles Sheriff Department
LBL	Lawrence Berkeley Laboratory, Berkeley, California
LC	liquid chromatography
LOD	limit of detection
LOI	letter(s) of instruction
NO _x	nitrogen oxide (s)

MR	multiple range
MRI	Midwest Research Institute, Kansas City, Kansas
MS	mass spectrometry (or mass spectrometer)
MSA	Mine Safety and Appliance Company
NA	not targeted for analysis <i>or</i> not applicable
NASA	National Aeronautical and Space Administration
NATICH	National Air Toxics Information Clearinghouse
NBS-SRM	National Bureau of Standards (now NIST)- Standard Reference Material
ND	no data <i>or</i> detection limit not determined
NEPA	National Environmental Policy Act
NF	not found in the sample matrix <i>or</i> not determined
NIST	National Institute of Science and Technology
nitro-PAH	nitropolycyclic aromatic hydrocarbons
NIOSH	National Institute for Occupational Safety and Health
NOSIH	Naval Ordnance Station, Indian Head, Maryland
NO _x	nitrogen oxides
NS	not sampled
OB	open burning
OB/OD	open burning/open detonation
OC	organic carbon
OD	open detonation
OGC	Oregon Graduate Center, Beaverton, Oregon
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbons
PANH	polycyclic aromatic nitrogen heterocycles
PAOH	polycyclic aromatic oxygen heterocycles
PCDD	polychlorinated dibenzodioxins
PCDF	polychlorinated dibenzofurans
PETN	pentaerythritol tetranitrate
PEP	propellants, explosives, and pyrotechnics
PIC	products of incomplete combustion
PICI/SIM	Positive ion chemical ionization/selective ion monitoring

PID	photoionization detector
PIP	product improvement program
PM	program manager
PMS	Particle Measuring Systems, Inc.
PUF	polyurethane foam
QA	quality assurance
QA/QC	quality assurance/quality control
QC	quality control
QAA	quality assurance agency
QAPP	quality assurance project plan
QAU	quality assurance unit
RCRA	Resource Conservation and Recovery Act
RDX	hexamethylenetrinitramine
RFD	Reno (Nevada) Fire Department
RIC	relative ion count
RSD	relative standard deviation
RTP	Research Triangle Park, North Carolina
SDPDA	Special Defense Property Disposal Account
SEM	scanning electron microscope/microscopy
SFC	supercritical fluid chromatography
SFC/MS	supercritical fluid chromatography/mass spectrometry
SF ₆	sulfur hexafluoride
SIM	selected-ion monitoring (or selective-ion monitoring)
SNL	Sandia National Laboratories, Albuquerque, New Mexico
SOP	standing operating procedures
SS	stainless steel
SSC	stainless steel canister
SSL	Sunset Laboratory, Forest Grove, Oregon
STEL	short-term exposure limit
STP	standard temperature and pressure (25°C and 760 torr)
TCD	thermal conductivity detector
TDP	test design plan

TEAD	U.S. Army Tooele Army Depot, Tooele, Utah
TECO	Thermo Electron Instruments (Company)
TECOM	U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland
THC	total hydrocarbon
TLV	threshold limit values
TNT	2,4,6-trinitrotoluene
TSC	technical steering committee
TSP	total suspended particulate
TW _α	time-weighted average
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland
UV	ultraviolet
VOC	volatile organic compounds
semi-VOST	semivolatile organic sampling train
VSDM	Volume Source Diffusion Model
XRF	ray fluorescence <i>or</i> X-ray fluorescence spectrometer

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APPENDIX B. SAMPLING AND DETERMINATION OF POLYCHLORINATED
DIBENZODIOXINS AND DIBENZOFURANS

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1. Introduction

This appendix describes the sample collection and analysis for atmospheric polychlorinated dibenzodioxins (PCDD) and polychlorinated dibenzofurans (PCDF) resulting from a test burn of composite fuel having the composition shown in Volume 2, Table 7.1. The test burn occurred on February 16, 1989 in the air building ("BB") at Sandia National Laboratories, Albuquerque, New Mexico. Samples were collected using high volume air samplers each of which was fitted with a quartz fiber filter followed by a polyurethane foam (PUF) sorbent cartridge. Analytical methodology applied to the subsequent extracts of the sampling media consisted of capillary column gas chromatography coupled to high resolution selected ion monitoring mass spectrometry.

2. Experimental

2.1 Sampling

Sampling was conducted using two General Metal Works PS-1 High Volume samplers provided by Battelle. Each sampler was fitted with a 20 cm by 25 cm quartz fiber filter followed by a polyurethane foam (PUF) plug contained in an Andersen glass cartridge. Sampling was conducted at a flow rate of 10.0 cfm (283 L/min) measured at a local temperature and pressure of 648 torr (864 mb), 13 °C. The filters were obtained from Pallflex Products Corporation, Putnam, CT, and prepared at Battelle prior to sampling by heating in a muffle furnace at 450 °C for a period of approximately 16 hours.

The PUF material was obtained from General Metal Works Company, Cleves, OH, and prepared prior to sampling by successive washes with acetone, toluene, and benzene followed by Soxhlet extraction with benzene for a period of approximately 24 hours. The PUF cartridges were inserted into Andersen glass sampling cylinders which previously had been treated in a muffle furnace under the same conditions as the filters. The filters were mounted and sealed in cassettes prior to transport to the test site. The PUF cartridges were each spiked with 10 ng of 1,2,3,4-TCDD- $^{13}\text{C}_{12}$ and individually placed in clean wide mouth screw cap jars with Teflon cap liners. Filter and PUF sampling media were hand carried to the test site by the Battelle sampling team.

The two General Metal Works samplers were tested at Battelle prior to shipment to the test site. Flow calibration of the samplers was conducted on-site at the Sandia BB using the manufacturer prescribed critical orifice procedure, on February 15, 1989. Four background samples (two each filter and PUF) were collected in the BB on February 16, 1989 beginning at 6:41 am, MST. The sampling rate of each sampler was 283 L/min with a sampling duration of 120 min. Sampling media were removed at the end of the background sampling, returned to their original shipping containers, and replaced with clean sampling media. Duplicate sampling at 283 L/min was initiated at 12:35 pm MST. Ignition of the 1 lb fuel sample occurred at 12:51 pm MST. Sampling was conducted for 120 minutes following ignition.

Samples returned to Battelle from the BB site consisted of two each filter and PUF from the background sampling, two each filter and PUF from the composite fuel burn sampling, and one each unused filter and PUF to serve as media travel blanks.

2.2 Sample Extraction and Analyte Enrichment

The samples were transferred to Soxhlet extraction thimbles and spiked with known amounts of the following isotope labelled internal standards:

<u>Internal Standard</u>	<u>Amount, ng</u>
2,3,7,8-tetrachlorodibenzo-p-dioxin- ¹³ C ₁₂ (2,3,7,8-tetra-CDD- ¹³ C ₁₂)	11.00
1,2,3,7,8-pentachlorodibenzo-p-dioxin- ¹³ C ₁₂ (1,2,3,7,8-penta-CDD- ¹³ C ₁₂)	10.55
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin- ¹³ C ₁₂ (1,2,3,6,7,8-hexa-CDD- ¹³ C ₁₂)	13.29
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin- ¹³ C ₁₂ (1,2,3,4,6,7,8-hepta-CDD- ¹³ C ₁₂)	8.74
Octachlorodibenzo-p-dioxin- ¹³ C ₁₂ (octa-CDD- ¹³ C ₁₂)	18.06
2,3,7,8-tetrachlorodibenzofuran- ¹³ C ₁₂ (2,3,7,8-tetra-CDF- ¹³ C ₁₂)	11.05
1,2,3,7,8-pentachlorodibenzofuran- ¹³ C ₁₂ (1,2,3,7,8-penta-CDF- ¹³ C ₁₂)	11.02
1,2,3,4,7,8-hexachlorodibenzofuran- ¹³ C ₁₂ (1,2,3,4,7,8-hexa-CDF- ¹³ C ₁₂)	10.36
1,2,3,4,6,7,8-heptachlorodibenzofuran- ¹³ C ₁₂ (1,2,3,6,7,8-hepta-CDF- ¹³ C ₁₂)	9.07

The above internal standards were added to serve as a basis for quantification of the native PCDD and PCDF sampled at the test site. Ten (10) ng of 1,2,3,4-TCDD-¹³C₁₂ was spiked in the PUF samples before sampling. The purpose of this isotope labelled spike was to verify the sample collection efficiency (absence of breakthrough) for each PUF cartridge. The filters were not spiked. Benzene was added to the extractors and the samples were extracted for 18 hours. The benzene extracts were then concentrated to approximately 5 mL using 3-stage Snyder columns.

The benzene extracts were transferred to multilayered silica gel columns containing activated silica gel, 44 percent concentrated sulfuric acid on silica gel, and 33 percent 1M sodium hydroxide on silica gel. The purpose of these columns was to remove acidic and basic

compounds from the extracts as well as to remove easily oxidized materials. The silica gel support provided a large surface area for contact with the sample extracts thus improving the cleanup efficiency. The PCDD/PCDF isomers were eluted from the columns using 70 mL of hexane and the entire eluates, including the original benzene extract volume, were collected. The benzene/hexane eluates were concentrated using a gentle stream of nitrogen gas and solvent exchanged into hexane. The hexane solutions were chromatographed through columns containing approximately 5 g of activated basic alumina using hexane:methylene chloride (97:3, v/v), and hexane:methylene chloride (1:1, v/v) as elution solvents. The 1:1 hexane:methylene chloride eluates were collected, concentrated to near dryness and diluted with 20 μ L of decane. Five ng of the recovery standard, 2,3,7,8-TCDD- $^{37}\text{Cl}_4$, was added to the extracts immediately prior to analysis. The purpose of this internal standard was to provide a mechanism for assessing the recovery of the nine isotope labelled internal standards added to each sample prior to extraction and cleanup.

2.3 Analysis

The extracts were analyzed and quantified for PCDD/PCDF using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The HRGC/HRMS consisted of a Carlo Erba Model 4160 gas chromatograph interfaced directly into the ion source of a VG Model 7070 high resolution mass spectrometer. The chromatographic column was a 60M DB-5 fused silica column using helium carrier gas at an average flow velocity of 30 cm/sec, measured at a column temperature of 160 °C. The mass spectrometer was operated in the electron impact (EI) ionization mode at a mass resolution of 9,000-12,000 (M/M, 10% valley definition). All HRGC/HRMS data were acquired by multiple-ion-detection (MID) using a VG Model 11-250J Data System. Operating conditions and the exact masses that were monitored are shown in Tables 1 and 2.

2.4 Quality Control

The operation of the HRGC/HRMS was evaluated with each set of samples by analyzing three standard mixtures of PCDD/PCDF isomers. The first mixture consisted of selected PCDD/PCDF isomers and was used to evaluate the stability of the chromatographic elution

windows. The second mixture, ten native PCDD/PCDF and eleven ^{13}C -labelled internal and recovery standards provided data to establish average response factors. The third mixture, five tetra-CDD isomers, was used to evaluate isomer resolution. The mass focus accuracy of the mass spectrometer was evaluated before each analytical determination by observing selected ion masses from perfluorokerosene (PFK). Computer assisted adjustments were made to the high voltage offset to correct for minor mass focus variations. Mass focus stability was assured by continuous monitoring, throughout each analysis, of a reference PFK "lock mass" to correct the mass spectrometer high voltage for any mass focus drift.

Native spike and method blank controls were processed during the extraction and cleanup of the samples. The native spike control was used to further evaluate the extraction and cleanup efficiency, while the laboratory method blank control was used to determine if the analytical results were the result of laboratory induced artifacts. The extractors associated with the native spike and method blank controls did not contain sampling media.

2.5 Recovery of Internal Standards

Recoveries of the PCDD/PCDF internal standards (2,3,7,8-tetra-CDD- $^{13}\text{C}_{12}$, 1,2,3,7,8-penta-CDD- $^{13}\text{C}_{12}$, 1,2,3,6,7,8-hexa-CDD- $^{13}\text{C}_{12}$, 1,2,3,4,6,7,8-hepta-CDD- $^{13}\text{C}_{12}$, octa-CDD- $^{13}\text{C}_{12}$, 2,3,7,8-tetra-CDF- $^{13}\text{C}_{12}$, 1,2,3,7,8-penta-CDF- $^{13}\text{C}_{12}$, 1,2,3,4,7,8-hexa-CDF- $^{13}\text{C}_{12}$, and 1,2,3,4,6,7,8-hepta-CDF- $^{13}\text{C}_{12}$) were calculated by comparison to the external standard (2,3,7,8-tetra-CDD- $^{37}\text{Cl}_4$), which was added following extraction. Recovery calculations are a measure of extraction efficiency and losses which may occur during clean-up and concentration.

The equation used to calculate the internal standard recoveries was:

$$\text{Recovery (\%)} = \frac{A_{is} \times Q_{rs} \times 100}{A_{rs} \times Q_{is} \times R_f}$$

Where:

- A_{is} = Sum of integrated areas for internal standard;
- Q_{rs} = Quantity of recovery standard in ng;
- Q_{is} = Quantity of internal standard in ng;
- A_{rs} = Sum of integrated areas for recovery standard, and;
- R_f = Average response factor of internal standard vs. recovery standard.

2.6 Quantification

The PCDD/PCDF isomers were quantified by comparing the sum of the two ions monitored for each native class to the sum of the two ions monitored for the corresponding isotopically labelled internal standard. Assuming the labelled PCDD/PCDF internal standards behave similarly to the native PCDD/PCDF in the sample, using the labelled internal standards for quantification compensates for recovery and extraction/clean-up efficiency. This is not always a good assumption since the internal standards are not environmentally incorporated in the sample matrix. The OCDD-¹³C₁₂ was used to quantify OCDF since no OCDF-¹³C₁₂ is available.

Experimental relative response factors were calculated from multiple analyses of a mixture which contained representatives of the tetrachloro- through octachloro-PCDD/PCDF congener classes. These response factors were included in all calculations used to quantify the data. The response factors were calculated using the sum of the two ions monitored for each congener class compared to the sum of the two ions monitored for the corresponding internal standard. The formula used for quantifying the PCDD/PCDF isomers was:

$$\text{Quantity/Sample} = \frac{A_c \times Q_{is} \times 1000}{A_{is} \times R_f}$$

Where:

- Quantity = Total quantity (pg) of target isomer or congener class;
- A_c = Sum of integrated areas for the target isomer or congener class;
- Q_{is} = Quantity of internal standard in ng;
- A_{is} = Total integrated areas for the internal standard;
- R_f = Response factor.

Each pair of resolved peaks in the selected-ion-current chromatograms was evaluated manually to determine if it met the criteria for a PCDD or PCDF isomer. By examining each pair of peaks separately, quantitative accuracy was improved over what is obtained when all of the peaks in a selected chromatographic window are averaged.

The criteria that were used to identify PCDD and PCDF isomers were:

- (1) Simultaneous responses at both ion masses;
- (2) Chlorine isotope ratio within ± 15 percent of the theoretical value;
- (3) Chromatographic retention times within windows determined from analyses of standard mixtures; and
- (4) Signal to noise ratio equal to or greater than 2.5 to 1.

The 2,3,7,8-tetra-CDD/tetra-CDF isomers and the octa-CDD included the additional criterion that they coeluted within ± 2 seconds of their isotopically labelled analogs.

A limit of detection (LOD) or maximum possible concentration was calculated for samples in which isomers of a particular chlorine congener class were not detected. The formula used for calculating the LOD was:

$$\text{LOD/Sample (pg)} = \frac{H_c \times Q_{is} \times 2.5 \times 1000}{H_{is} \times R_f}$$

where:

LOD = Single isomer limits of detection (pg) for a congener class;

H_c = Sum of the peak heights of congener class isomer;

Q_{is} = Quantity of internal standard (ng);

H_{is} = Sum of the peak height of internal standard;

R_f = Response factor.

3. Discussion of Results

Low but comparable levels of hexa-, hepta-, and octa-chlorodibenzofuran were detected in the duplicate burn filter samples (Table 3), at levels significantly higher than those observed in the method blank. These data are not corrected for background samples. The laboratory method blank contains tetra- through hexachlorodibenzofurans. This contamination is the result of laboratory procedures which may have resulted in the small quantity of penta-CDF observed on one burn filter (13F). It is unlikely that the tetra-CDF resulting in contamination of the method blank contributed to the small quantity of tetra-CDF observed in the burn filter 13F. The tetra-CDF observed in this burn filter consists of one isomer (2,3,7,8-TCDF), and this isomer was not observed in the method blank.

Octachlorodibenzo-p-dioxin (OCDD) was detected in several samples including a background air filter (Table 4). A small quantity of heptachlorodibenzo-p-dioxin was detected on one of the burn filters as well as the corresponding PUF sample. No other dioxins were detected in any sample. OCDD is a common contaminant in many matrices and the low level found

here does not represent an unusual event. Because no other dioxins were detected, a direct link between the combustion source and the air samples is difficult to establish.

The internal standards were well recovered in all the samples (Table 5) which indicates that the extraction and preparation of the samples was performed efficiently. Also, the native spike sample was well recovered which suggests confidence in the accuracy of the method. The pre-sampling PUF spike (1,2,3,4-TCDD-13C) was over-recovered in each of the four PUF samples analyzed. Percent recoveries (not shown in Table 5) were 204, 239, 155, and 196 percent for samples 11P, 13P, 14P, and 15P respectively. These values are a factor of two greater than expected based upon previous experience with PUF sampling for these compounds. A detailed examination of laboratory procedures used with the OB/OD samples has failed to provide evidence to support spiking of the PUF samples at twice the expected level. We must therefore conclude that the PUF sampling efficiency for PCDD and PCDF cannot be substantiated for these particular samples.

In order to evaluate the concentrations of the analytes in the bang box atmosphere immediately following the test burn, the actual sampling volume of the original bang box atmosphere must take into account the exponential dilution of the air within the bang box during the 120 minute sampling period. With constant physical sampling rate, the rate at which the original atmospheric composition is sampled decreases exponentially with time as:

$$R(t) = R_0 \exp(-kt)$$

where: $R(t)$ is the instantaneous sampling rate at time t of original atmosphere (at $t=0$, immediately following the burn), and R_0 is the physical sampling rate of 283 L/min. The bang box atmosphere half-life, provided by Dr. H. Smith Broadbent, was 21.87 min. during the composite fuel burn test. This allows evaluation of the dilution constant k in the above expression as:

$$k = \frac{-\ln(0.5)}{21.87} = 0.03169 \text{ min}^{-1}$$

Integration of the above exponential rate equation above over the interval 0-120 minutes yields the volume (V) of original post burn atmosphere (at $t=0$) sampled as:

$$v = \frac{R_0 \times (1 - \exp(-120k))}{k} = 8730 \text{ L} = 8.73 \text{ m}^3$$

Table 6 provides the PCDD results in concentration units of pg/m^3 while Table 7 provides the PCDF results in the same concentration units, based on the original atmosphere sampling volume of 8.73 m^3 , at 648 torr (864 mb), 13°C . No correction is made for the background sampling. These results assume that the outdoor air infiltrating the bang box is free of any PCDD/PCDF contamination, native PCDD/PCDF loss to the bang box structure is insignificant over the sampling interval, and the time required to complete the burn (approximately 5 seconds) as well as the time required to achieve atmospheric homogeneity at $t = 0$ is insignificant. The LODs provided in Tables 6 and 7 (values in parentheses) are expressed in the same concentration units (pg/m^3). The LODs for 2,3,7,8-TCDD, shown in Table 7, are significantly higher than the LOD ($0.24 \text{ pg}/\text{m}^3$) demonstrated during Battelle's urban air sampling in Columbus Ohio (S.A. Edgerton, J.M. Czuczwa, J.D. Rench "Ambient Air Concentrations of Polychlorinated-p-Dioxins and Dibenzofurans in Ohio: Sources and Health Risk Assessment", submitted to Chemosphere). The elevated LODs in the bang box experiment are due to the small volume of atmosphere at $t = 0$ that was actually sampled. An increase in sampling time would not have significantly improved the LODs. Lower LODs

may be achievable by the use of multiple parallel samples that are subsequently pooled, or testing in an enclosure having a significantly smaller leak rate.

TABLE 1. HRGC/HRMS OPERATING PARAMETERS

Mass Resolution definition)	9000-12000 (M/M, 10% valley
Electron Energy	70 eV
Accelerating Voltage (7070E)	4000 volts (7070H) or 6000 volts
Source Temperature	225-250 °C
Preamplifier Gain	10^{-7} amp/volt
Electron Multiplier Gain	-10^8
Column	DB-5 60M
Transfer Line Temperature	300 °C
Injector Temperature	300 °C
Column Temperature - Initial	160 °C
Column Temperature - Program	10 °C/min to 225 °C hold for 40 min, then 15 °C/min to 320 °C hold for 20 min.
Carrier Gas	Helium
Flow Velocity	-30 cm/sec
Injection Mode	Splitless

TABLE 2. EXACT MASSES USED FOR THE DETERMINATION OF PCDD AND PCDF

Compound	Accurate Mass		Theoretical Isotope Ratio Mass 1/Mass 2
	Mass 1	Mass 2	
Tetrachlorodibenzo-p-dioxins	319.8965	321.8936	0.77
Tetrachlorodibenzofurans	303.9016	305.8987	0.77
Pentachlorodibenzo-p-dioxins	355.8546	357.8517	1.54
Pentachlorodibenzofurans	339.8597	341.8567	1.54
Hexachlorodibenzo-p-dioxins	389.8156	391.8127	1.23
Hexachlorodibenzofurans	373.8207	375.8178	1.23
Heptachlorodibenzo-p-dioxins	423.7766	425.7737	1.03
Heptachlorodibenzofurans	407.7817	409.7788	1.03
Octachlorodibenzo-p-dioxins	457.7377	459.7347	0.88
Octachlorodibenzofurans	441.7428	443.7398	0.88
Tetrachlorodibenzo-p-dioxin- ¹³ C ₁₂	331.9367	333.9338	0.77
Tetrachlorodibenzofuran- ¹³ C ₁₂	315.9418	317.9389	0.77
Pentachlorodibenzo-p-dioxin- ¹³ C ₁₂	367.8948	369.8918	1.54
Pentachlorodibenzofuran- ¹³ C ₁₂	351.8999	353.8969	1.54
Hexachlorodibenzo-p-dioxin- ¹³ C ₁₂	401.8558	403.8529	1.23
Hexachlorodibenzofuran- ¹³ C ₁₂	385.8609	387.8580	1.23
Heptachlorodibenzo-p-dioxin- ¹³ C ₁₂	435.8168	437.8139	1.03
Heptachlorodibenzofuran- ¹³ C ₁₂	419.8219	421.8190	1.03
Octachlorodibenzo-p-dioxin- ¹³ C ₁₂	469.7779	471.7749	0.88
Tetrachlorodibenzo-p-dioxin- ³⁷ Cl ₄	327.8847	--	--

TABLE 3. PCDF RESULTS FOR OBOD SAMPLES (pg/sample)^a

Sample Number	2,3,7,8-TETRA -CDF	TOTAL TETRA -CDF	TOTAL PENTA -CDF	TOTAL HEXA -CDF	TOTAL HEPTA -CDF	OCTA -CDF
SNL-16-02-11P	(4.0)	(4.0)	(4.1)	(2.4)	(1.5)	(12.0)
SNL-16-02-13P	(16.3)	(16.3)	(25.2)	(9.0)	(11.7)	(52.7)
SNL-16-02-14P	(24.2)	(24.2)	(12.3)	(10.6)	(8.4)	(98.2)
SNL-16-02-15P	(6.8)	(6.8)	(6.0)	(4.1)	(1.2)	(8.5)
SNL-16-02-11F	(42.2)	(42.2)	(6.6)	(6.6)	(15.4)	(37.7)
SNL-16-02-13F	23.0	23.0	31.3	134	199	274
SNL-16-02-14F	(56.7)	(56.7)	(31.2)	145	245	282
SNL-16-02-15F	(26.3)	(26.3)	(5.6)	(7.2)	(7.7)	(19.4)
Native Spike ^b	5320	5320	2760	4280	3560	11,200
Method Blank	(38.4)	322	98.6	31.2	(24.1)	(27.2)

^aParenttheses indicate analyte not detected; value within parentheses represents analytical detection limit.^bNative spike is fortified before extraction with the following: 5700 pg 2,3,7,8-TCDF, 5700 pg total tetra-CDF, 2930 pg total penta-CDF; 4600 pg total hexa-CDF, 3700 pg total hepta-CDF, 12500 pg octa-CDF.^cNanograms.

TABLE 4. PCDD RESULTS FOR OBOD SAMPLES (pg/sample)^a

Sample Number	2,3,7,8-TETRA-CDD	TOTAL TETRA-CDD	TOTAL PENTA-CDD	TOTAL HEXA-CDD	TOTAL HEPTA-CDD	OCTA-CDD
SNL-16-02-11P	(11.8)	(11.8)	(10.0)	(2.8)	(6.1)	(14.5)
SNL-16-02-13P	(216)	(216)	(47.6)	(14.0)	9.7	90.0
SNL-16-02-14P	(53.8)	(53.8)	(29.0)	(21.5)	(33.0)	73.6
SNL-16-02-15P	(8.3)	(8.3)	(31.6)	(7.9)	(8.3)	(26.6)
SNL-16-02-11F	(92.8)	(92.8)	(34.2)	(28.2)	(22.9)	52.6
SNL-16-02-13F	(46.7)	(46.7)	(83.6)	(12.4)	13.0	47.4
SNL-16-02-14F	(54.9)	(54.9)	(85.8)	(15.1)	(40.5)	43.4
SNL-16-02-15F	(44.4)	(44.4)	(30.3)	(15.7)	(30.5)	(80.4)
Native Spike ^b	5050	5050	5880	4560	3300	3790
Method Blank	(105)	(105)	(143)	(19.3)	(28.8)	(77.6)

^aParentheses indicate analyte not detected; value within parentheses represents analytical detection limit.

^bNative spike is fortified before extraction with the following: 5480 pg 2378-tetra-CDD, 5480 pg total tetra-CDD; 6750 pg total penta-CDD; 5000 pg total hexa-CDD; 3290 pg total hepta-CDD; and 3310 pg octa-CDD.

TABLE 5. RECOVERIES OF PCDD/PCDF INTERNAL STANDARDS (%)

Sample Number	TCDF -13C	TCDD -13C	PCDF -13C	PCDD -13C	HXCDF -13C	HXCDD -13C	HPCDF -13C	HPCDD -13C	OCDD -13C
SNL-16-02-11P	114	111	96	96	94	99	97	110	123
SNL-16-02-13P	140	136	117	126	91	98	99	111	107
SNL-16-02-14P	110	95	125	140	88	91	88	96	97
SNL-16-02-15P	103	125	83	37	82	93	94	92	93
SNL-16-02-11F	124	101	142	51	100	100	98	110	117
SNL-16-02-13F	111	108	93	99	82	87	82	90	100
SNL-16-02-14F	108	100	82	87	69	71	63	72	73
SNL-16-02-15F	99	90	128	128	92	95	97	107	115
Native Spike	102	95	118	125	82	83	95	90	86
Method Blank	101	92	121	104	102	101	115	130	129

TABLE 6. PCDF RESULTS FOR OBOD SAMPLES (pg/m³)^a

Sample Number	2,3,7,8- TETRA -CDF	TOTAL TETRA -CDF	TOTAL PENTA -CDF	TOTAL HEXA -CDF	TOTAL HEPTA -CDF	OCTA -CDF
SNL-16-02-11P	(0.46)	(0.46)	(0.47)	(0.28)	(0.17)	(1.38)
SNL-16-02-13P	(1.87)	(1.87)	(2.88)	(1.03)	(1.34)	(6.03)
SNL-16-02-14P	(2.77)	(2.77)	(1.4)	(1.21)	(0.96)	(11.3)
SNL-16-02-15P	(0.78)	(0.78)	(0.69)	(0.46)	(0.14)	(0.97)
SNL-16-02-11F	(4.84)	(4.84)	(0.76)	(0.75)	(1.77)	(4.32)
SNL-16-02-13F	2.64	2.64	3.58	15.3	22.8	31.4
SNL-16-02-14F	(6.50)	(6.50)	(3.57)	16.6	28.1	32.3
SNL-16-02-15F	(3.01)	(3.01)	(0.64)	(0.82)	(0.89)	(2.22)

^aParentheses indicate analyte not detected; value within parentheses represents analytical detection limit.

TABLE 7. PCDD RESULTS FOR 080D SAMPLES (pg/m³)^a

Sample Number	2,3,7,8- TETRA -CDD	TOTAL TETRA -CDD	TOTAL PENTA -CDD	TOTAL HEXA -CDD	TOTAL HEPTA -CDD	OCTA -CDD
SNL-16-02-11P	(1.36)	(1.36)	(1.15)	(0.32)	(0.70)	(1.66)
SNL-16-02-13P	(24.8)	(24.8)	(5.45)	(1.60)	1.11	10.3
SNL-16-02-14P	(6.16)	(6.16)	(3.32)	(2.47)	(3.78)	8.43
SNL-16-02-15P	(0.95)	(0.95)	(3.62)	(0.90)	(0.95)	(3.04)
SNL-16-02-11F	(10.6)	(10.6)	(3.92)	(3.23)	(2.63)	6.03
SNL-16-02-13F	(5.35)	(5.35)	(9.58)	(1.42)	1.49	5.43
SNL-16-02-14F	(6.28)	(6.28)	(9.83)	(1.73)	(4.64)	4.97
SNL-16-02-15F	(5.09)	(5.09)	(3.47)	(1.79)	(3.49)	(9.21)

^aParentheses indicate analyte not detected; value within parentheses represents analytical detection limit.

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